THE NICKEL BULLETIN

A SUMMARY OF CURRENT INFORMATION ON NICKEL

RECEIVED
SEP 8 1953
LIBRARY

APRIL, 1953

SIMPLE TESTS FOR IDENTIFYING SOME NICKEL ALLOYS AND OTHER METALS *page* 48

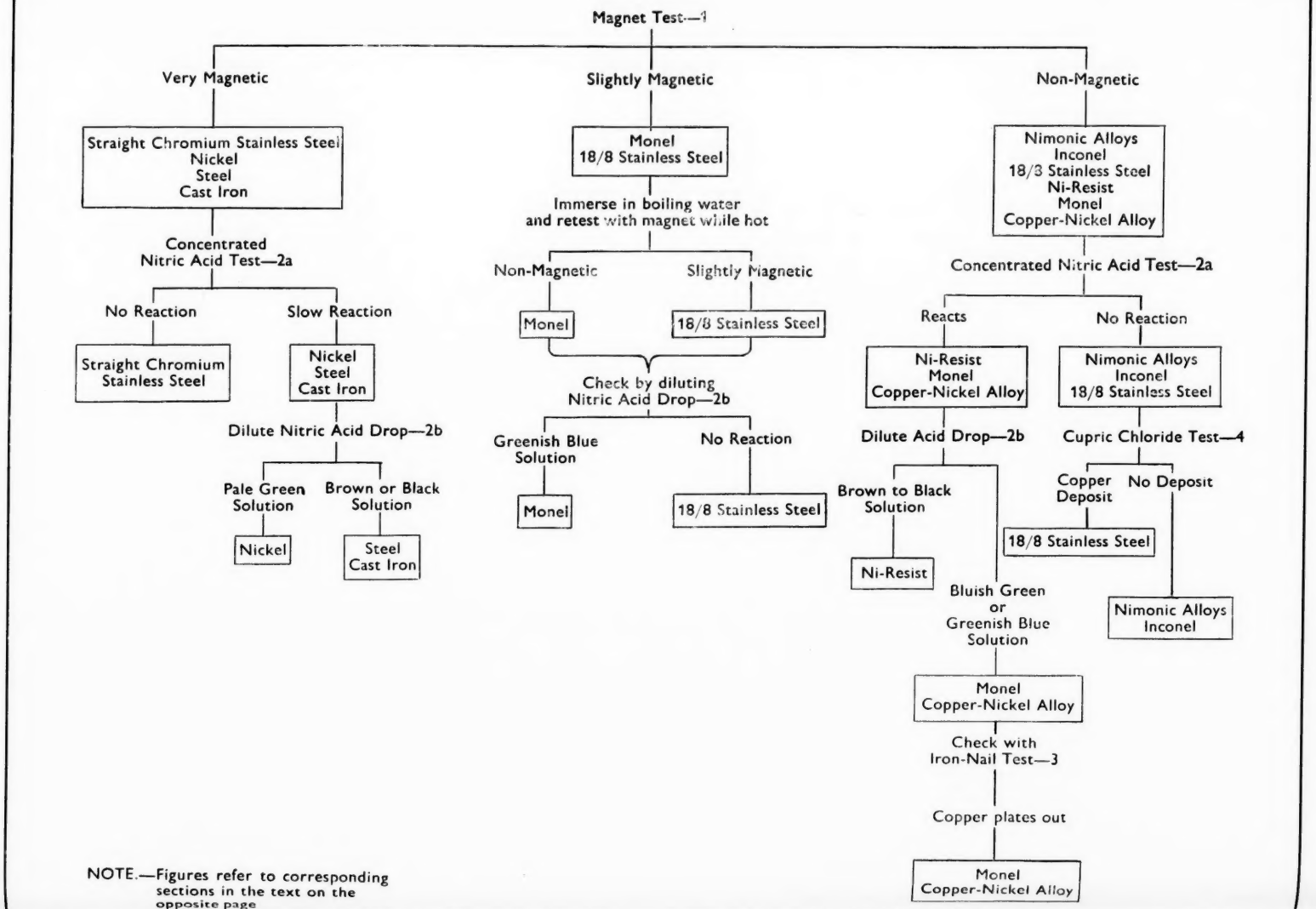
	Page
General	
Metals Specifications Handbook	50
Electrolytic 'Thinning' of Metals and Alloys	50
Nickel	
Conservation of Nickel: Modified Regulations	50
Raney-type Nickel Catalysts	51
Nickel Carbide	51
Welding of Nickel-Clad Steel by Shielded Metal-Arc Process	51
Electrodeposition and Other Coating Methods	
Electroplating Research in Great Britain	51
Effect of Temperature on Cathode Potential in Electrodeposition of Nickel	52
Nickel Plating of Aluminium-base Materials	52
Determination of Total Sulphates in Nickel-Plating Baths	53
Non-Ferrous Alloys	
Nickel-containing Coinage: Report of Royal Mint	53
Monel Stays in Standard Locomotives	54
Nickel-Titanium Alloys: Constitution	54
Welding of 90-10 Cupro-Nickel Alloys	54
Estimation of Silicon in Nickel Alloys	54
Nickel-Iron Alloys	
Low-Expansion Nickel-Iron Alloys: Properties and Treatment	55
Determination of Sulphur in Nickel-Iron Alloys	55
Constructional Steels	
Line Broadening of Martensite in Nickel Steels	55
Machinability Tests on Cast and Wrought Nickel-Alloy Steels	56
Thermal Stabilization of Austenite in Iron-Carbon-Nickel Alloys	56
Physical Constants of Steels at Elevated Temperatures	56
Spectrographic Analysis of Low-Alloy Steels	57

	Page
Heat- and Corrosion-Resisting Alloys	
High-Temperature Materials: Typical Compositions and Properties	57
Creep Characteristics of Nickel	58
Influence of Boron on Oxidation-Resistance of Chromium-Nickel-Cobalt-Iron Alloys	59
Thermal-Shock Tests on Austenitic and Ferritic Materials	59
Reactions between Refractory Oxides and Metals at High Temperatures	60
Ceramic Coating of Heat- and Corrosion-Resisting Materials	60
Creep Testing of Nimonic Alloys	60
Subsurface Porosity caused by High-Temperature Corrosion	60
Contamination of Condensate in Heat Exchangers: Tests on Nickel-containing Alloys	61
Resistance of Cast Iron-Chromium-Nickel Alloys to Corrosion by Flue Gases	62
Welding of High-Temperature Materials for Gas-Turbine Parts	63
X-Ray Fluorescent Analysis of High-Temperature Alloys	63
Influence of Hydrogen on Mechanical Properties of High-Alloy Nickel-containing Steels	64
Chi Phase in Molybdenum-containing Stainless Steels	65
Structural Hardening of Austenitic-Ferritic Steels: Utilization of Sigma Phase	65
Influence of Cold Working on Magnetic Properties of Austenitic Chromium-Nickel Steels	65
Stress-Corrosion Cracking of Chromium-Nickel Austenitic Steels	66
Corrosion-Resisting Materials in Plant of Celanese Corporation of America	67
Soil-Corrosion Tests on Irons and Steels	67
Pickling of Austenitic Chromium-Nickel Steels	68
Copper-Sulphate Sulphuric-Acid Test for Stainless Steels	68
Patents	68

JWC
PT
GPG
CB
HNH
A Z
OH

VOLUME 26 NUMBER 4

THE MOND NICKEL COMPANY LTD • SUNDERLAND HOUSE, CURZON STREET, LONDON, W.1
Telephone:- Grosvenor 4131



NOTE.—Figures refer to corresponding sections in the text on the opposite page

SIMPLE TESTS FOR IDENTIFYING SOME NICKEL ALLOYS AND OTHER METALS

RAPID identification of common metals and alloys is a necessary stage for the segregation of materials and for the optimum salvage of scrap.

This outline offers simple testing procedures which can be used in the laboratory but are primarily for use in the shop or scrap yard. Before testing, the surface of the materials being examined should be thoroughly cleaned with a file or with emery cloth to remove dirt, grease or corrosion products, metallic coatings applied by electrodeposition, spraying or galvanizing, or any casting skin. Care should also be taken to see that the specimen is homogeneous and not bi-metallic, as, for example, nickel-clad or stainless-clad scrap.

1. Magnetic Tests

Bring the magnet into contact with the specimen to be tested, taking care that there is no magnetic material in the vicinity. The specimen may or may not be attracted to the magnet.

In cases of doubt, and especially when the material appears to be non-magnetic, a more sensitive method of testing should be used:—

(*a*) Balance the magnet or the specimen on the edge of a bench or desk. Then bring the specimen close to the magnet, or *vice versa*. If the material is magnetic the balance will be upset and a movement of either the magnet or the specimen will be seen.

(*b*) Another method is to suspend the magnet on a string and bring it close to the specimen. If the specimen is even slightly magnetic a slight movement will be observed.

Reactions to the magnet test may be graded as:—

Non-Magnetic. Nimonic alloys, Inconel, Ni-Resist, copper-nickel alloys containing less than 60 per cent. of nickel (e.g., nickel silvers), 'K' Monel, chromium-nickel stainless steel.

Slightly Magnetic. Chromium-nickel stainless steel, Monel.

Strongly Magnetic. Ordinary steel or cast iron, chromium stainless steel or nickel.

Monel is usually but not always slightly magnetic at atmospheric temperatures. When only a magnet is available, Monel can be identified by the following test:—

Monel which appears to be non-magnetic at ordinary temperatures may become magnetic while dipped in ice water or in a freezing mixture. (Other copper-nickel alloys of lower nickel content do not become magnetic.) Monel that is magnetic at ordinary temperatures loses its magnetism while dipped in boiling water.

'K' Monel is always non-magnetic even when cooled. Chemical analysis is required to distinguish 'K' Monel from copper-nickel alloys of lower nickel content.

Certain grades of chromium-nickel corrosion-resisting steel of the 18/8 type may be either slightly magnetic in the annealed condition and may vary from non-magnetic to slightly magnetic, or even quite appreciably magnetic, if severely cold-worked, but will never be as strongly magnetic as the straight chromium stainless steel. The magnetic test can, therefore, be relied upon to distinguish between these two materials.

2. Nitric Acid Tests

(*a*) Clean the test surface so that the acid will come into direct contact with the metal.
Apply one or two drops of concentrated nitric acid to the clean metal surface.
Allow the acid to remain in contact with the metal for a minute or two, noting any reaction which takes place.

(*b*) Dilute the acid by adding three or four drops of water, one drop at a time. If the solution turns green or blue leave it undisturbed for the nail test.

3. Copper Test with Iron Nail

If the nitric acid test forms green or blue solution make the following test for copper:—

Rub a clean iron nail in the coloured solution on the metal surface, keeping the nail in contact with the specimen.

If the alloy contains copper, there will be a deposit of copper either on the nail or on the surface of the metal under the acid solution.

Add a drop or two of water as the nail is rubbed around to prevent the deposited copper from re-dissolving in the acid.

4. Drop Test with Cupric Chloride Solution

This procedure serves only to distinguish Inconel and Nimonic alloys from the 18/8 type of stainless steel and is used to supplement but not to replace the magnetic and nitric acid tests.

Apply one drop of the solution of cupric chloride in hydrochloric acid to the cleaned metal surface and leave it in contact with the metal for two minutes.

At the end of two minutes, add three or four drops of distilled water slowly, one drop at a time.

Wash the solution off the metal.

If the sample is of the 18/8 type of stainless steel, the copper in the cupric chloride solution will be deposited on the metal surface, appearing as a copper-coloured spot. If the sample is Inconel there will be only a white spot where the testing solution was applied.

MATERIALS REQUIRED

(1) A good magnet.
(2) Pure concentrated nitric acid.
(3) Clean water, preferably distilled, for diluting the solutions.
(4) A solution of 10 g. of cupric chloride ($CuCl_2.H_2O$) in 100 c.c. of concentrated hydrochloric acid.
(5) Droppers for applying test solutions and water.
(6) Clean iron or steel nails for testing for copper.

Monel
Copper-Nickel Alloy

NOTE.—Figures refer to corresponding sections in the text on the opposite page

ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Metals Specifications Handbook

'Steel's Specifications Handbook. Cross Index of Chemically Equivalent Specifications and Identification Code for Ferrous and Non-Ferrous Alloys.' *Published by 'Steel'*, Cleveland, Ohio, 1952; 405 pp.

This handbook is based on a cross index of U.S. specifications compiled under the direction of the Office of Standardization, Department of Defense, Washington. It constitutes the most comprehensive volume of this type yet published. The work was carried out by a joint U.S. Army-Navy-Air Force group, in co-operation with the engineering staff of General Motors Corporation.

The handbook is divided into two parts: in Part I groups of specifications for materials of similar chemical composition are arranged in numerical order according to a code system, details of which are given: in Part II all specifications are listed according to the issuing agencies. The coverage provided by the volume, from the point of view of materials and standardizing bodies, is indicated below:—

Part I. Materials for which Specifications are listed

Ferrous alloys (plain and alloy steels, including stainless and heat-resisting steels, nickel-iron-base alloys, and ferrous welding rods); copper and copper-base alloys; nickel and nickel-base alloys; cobalt-base alloys; lead, tin and their alloys; silver and silver-base alloys; aluminium and aluminium-base alloys; magnesium and magnesium-base alloys; zinc and zinc-base alloys; bismuth- and cadmium-base alloys; antimony; platinum.

Part II. Standardizing Bodies whose Specifications are included

U.S. Army; Aeronautical Standards Group; U.S. Air Force; U.S. Federal Specifications Board; American Iron and Steel Institute; U.S. Munitions Board Standards Agency, covering Military (MIL) and Joint Army-Navy (JAN) specifications; Society of Automotive Engineers (including both general and Aeronautical Materials Standards series); American Society for Testing Materials. In a supplementary section of Part II, designations of products used by aluminium producers are correlated with the standard code-number system used in the Handbook. In this connexion details are given of the products of Aluminum Company of America, Reynolds Metals Company, and Kaiser Aluminum and Chemical Corporation.

An index provides access from type to code-group classification.

Physical Constants of Steels at Elevated Temperatures

See abstract on p. 56.

Electrolytic 'Thinning' of Metals and Alloys

I. EPELBOIN: 'Drastic Thinning Down (of Metal Strip and Film) by Electrolytic Removal of Metal, and Applications of the Process.'

Revue de Métallurgie, 1952, vol. 49, Dec., pp. 863-74.

The author describes fundamental investigation of the process of electrolytic polishing and metal removal, made in connexion with a study of the variation of physical properties of thin strip and foil of metals and alloys as a function of thickness. The first portion of the paper is concerned with the electrical conditions obtaining in electrolytic polishing, and this is followed by a detailed discussion of the factors involved in choice of composition for electropolishing solutions, and details of compositions of baths suitable for polishing of various metals and alloys, including nickel, and cobalt-nickel and iron-nickel alloys.

The author then deals at length with the application of electropolishing methods for the purpose of thinning-down metallic specimens : technique is fully described and apparatus is illustrated. The final section of the paper contains a report of specific results obtained by the process of thinning-down, on thin strip or film of nickel, a complex alloy of Mu-metal type (containing nickel 67, iron 25, molybdenum 4, copper 4, per cent.) and Supermalloy (molybdenum-containing nickel-iron alloy), on which materials it was desired to determine certain magnetic and physical properties as a function of thickness, in material of extremely thin gauge. In discussion, attention was directed by P. A. JACQUET to commercial application of the electrolytic thinning-down method, as practised in the production of very thin stainless-steel wire (see *Metal Progress*, 1951, vol. 59, June, pp. 795-7; *Nickel Bulletin*, 1951, vol. 24, No. 8, p. 184).

NICKEL

Conservation of Nickel: Modified Regulations

MINISTER OF SUPPLY: 'The Nickel Prohibited Uses (Minister of Supply) (Amendment) Order 1953.' *Statutory Instruments*, 1953, No. 265; Feb. 20, 1953. Board of Trade, 'The Nickel Prohibited Uses (Board of Trade) (Amendment No. 2) Order, 1953.' *Statutory Instruments*, 1953, No. 266; Feb. 20, 1953.

These Orders, which modify the regulations issued in Minister of Supply and Board of Trade Orders of

1951, cover the following broad changes in permitted use of nickel-containing materials:—

(*a*) the use of nickel and nickel alloys for welding is freed from control.

(*b*) the use of stainless steel containing not more than 8 per cent. by weight of nickel is permitted in the manufacture of hospital hollow-ware, and in certain parts of urns (containers) in contact with liquid.

(*c*) the use of nickel silver containing not more than 15 per cent. by weight of nickel is permitted in the manufacture of table hollow-ware, harness, and saddlery.

(*d*) the nickel plating of hat pins, safety pins, file and loose-leaf fittings, and certain parts of ball-point pens and propelling pencils is freed from control.

Raney-Type Nickel Catalysts

L. M. KEFELI and S. L. LEL'CHUK: 'Structure of Skeleton Catalysts.' *Doklady Akad. Nauk. S.S.S.R.*, 1952, vol. 84, pp. 285-8; *Chemical Abstracts*, 1952, vol. 46, Nov. 25, p. 10,822.

'When the soluble component is leached out from a binary alloy, the remaining metal undergoes re-grouping into its characteristic lattice, e.g., after aluminium is leached out from the rhombohedral Ni_2Al_3 the skeleton nickel becomes cubic. On incomplete leaching only the unchanged Ni_2Al_3 retains its rhombohedral lattice, but all the nickel is cubic.' The authors discuss this finding (resulting from their own work) in the light of other studies which have been reported on the structure of completely and incompletely leached catalyst material. For summary of views of the respective investigators, see *Chemical Abstracts, loc. cit.*

N. P. KEIER: 'Rôle of the Different Adsorption Centres in the Hydrogenation of Acetylene on Metallic Nickel. I. Investigation of the Active Surface of Nickel with the Aid of the Differential Isotopic Method.' *Izvest. Akad. Nauk. S.S.S.R., Otdel. Khim. Nauk*, 1952, pp. 616-26; *Chemical Abstracts*, 1952, vol. 46, Nov. 25, pp. 10,820-1.

Desorption and catalytic reactions of acetylene on nickel were investigated by following the acetylene by portions, one of which was tagged with radioactive C_{14} as tracer. The desorbed gas or the gaseous reaction products were analyzed for C_{14} by radioactivity measurements. The catalyst used was made by leaching a 50-50 nickel-aluminium alloy. A detailed summary of the observations made is given in *Chemical Abstracts, loc. cit.*

Nickel Carbide

G. MEYER and F. E. C. SCHEFFER: 'The Properties of Nickel Carbide.' *Jnl. Amer. Chemical Soc.*, 1953, vol. 75, Jan. 20, p. 486.

Short note discussing results published by Browning and Emmett, which were not in agreement with earlier data published by Scheffer. The characteristics of the carbides examined by Browning and Emmett, by Scheffer, and by Bahr and Bahr, are briefly reviewed.

Metals Specifications Handbook

See abstract on p. 50.

Creep Characteristics of Nickel

See abstract on p. 58.

Welding of Nickel-Clad Steel by Shielded Metal-Arc Process

R. G. LYALL: 'Nickel-Clad Steels Welded by Shielded Metal-Arc Process.' *Iron Age*, 1953, vol. 171, Jan. 15, pp. 102-3.

This short article describes the application of the Aircomatic (inert-gas-shielded metal-arc) process in the production of evaporators for the alkali industry, in which nickel and $\frac{1}{2}$-in. thick 20 per cent. clad nickel-clad steel are used. Seams in the clad plate are welded from the clad side. Commercially pure nickel welding wire, $\frac{1}{16}$-in. dia., is deposited under a protective blanket of argon, flowing at the rate of 60-80 cu.-ft. per hour. Wire feed is 250 in. per minute for 20 per cent. clad plate, and 210 in. per minute for solid nickel plate. Reverse-polarity direct current is used, at 275-300 amp. Joint preparation from the outside is a 60° single bevel. The use of this process minimizes iron pick-up in welding and saves time in cleaning welds between passes, due to the fact that the welding wire is not coated. Multipass welding can thus be carried out without interruption. Joint detail between tube and shell plate is diagrammatically illustrated.

Electrolytic 'Thinning' of Metals and Alloys

See abstract on p. 50.

Estimation of Silicon in Nickel Alloys

See abstract on p. 54.

ELECTRODEPOSITION AND OTHER COATING METHODS

Electroplating Research in Great Britain

G. L. J. BAILEY: 'A Review of Co-operative Electroplating Research in Britain.' *Sheet Metal Industries*, 1953, vol. 30, Mar., pp. 213-16, 236.

This review traces the history of co-operative research from the work sponsored (at first directly) by the Department of Scientific and Industrial Research at the Research Department, Woolwich (1925-1937), to later collaboration between the British Non-Ferrous Metals Research Association and Woolwich, and, finally, to the setting up of an electroplating research and development department within the B.N.F.M.R.A. Some account is given of the methods which have been used to make the results of research available to the practical plater, and to encourage the collaboration of the industry.

The final section of the review briefly surveys some of the highlights of research results. Specific reference is made to the development of the B.N.F. jet test for

measurement of local thickness, and the vibrating-hammer adhesion test evolved at Woolwich. Attention is drawn to the wide range of fundamental research covered at Woolwich, including studies on influence of bath composition on pitting, porosity, stress, and metal distribution; the mechanism of formation of bright deposits, and their characteristics. It is emphasized that the practical benefits of much of this work are today being enjoyed and practically utilized, in most plating shops in the country.

In connexion with current requirements and future developments, attention is directed to further studies on porosity which are being carried out by the British Non-Ferrous Metals Research Association laboratory, to investigations on hard chromium deposits (with special reference to elimination of cracking), to research on electropolishing and the scope for use of this method in preparation of metallic surfaces for plating, and to ad-hoc work which has been carried out to assist industry in the most efficient utilization of the supplies of nickel available during the period of control and restriction.

Conservation of Nickel: Modified Regulations Relating to Nickel Plating

See abstract on p. 50.

Effect of Temperature on Cathode Potential in Electrodeposition of Nickel

D. R. TURNER: 'The Effect of Temperature on the Cathode Potential during Nickel Plating.'
Jnl. Electrochemical Soc., 1953, vol. 100, Jan., pp. 15-21.

The character of metal electrodeposits is controlled primarily by the nature of the electrolyte immediately adjacent to the cathode surface. For optimum results with a particular process, it is usually necessary to adjust the concentration of the bath constituents, the pH, the current density, and the temperature, to specific ranges. Each of these factors has some influence on the composition of the electrolyte at the metal-solution interface, and each affects the cathode potential. No extensive study had previously been made of the effect of temperature on cathode potential.

The author gives a detailed description (with drawing) of the experimental cell, and of deposition procedure. The bath used was of the Watts type, containing (g./L.) nickel sulphate 240, nickel chloride 38, boric acid 38. It was prepared from C.P. chemicals, and was treated in the standard manner for removal of organic and inorganic impurities. Such treatment included filtering at a pH of 5·0, treating with activated carbon, and electrolyzing 2 amp.-hr./L. of solution. The final pH adjustment was to 3·0.

The effect of temperature on cathode potential was studied at current densities ranging from 0·4 to 100 ma./cm.^{2}, and at temperatures of 20°-90°C. It was found that the cathode potential-temperature relation is linear, with sharp change in slope at about −0·42 volt. The E_c—T straight lines above −0·42 volt, when extrapolated towards higher temperatures, converge at about 0 volt and 165°C. Although the convergence to a common point would appear to be significant, especially at 0 volt, the author was unable to find any explanation. An empirical equation was developed from the data, in which the cathode potential is proportional to the square root of the current density:—

$$E_c = 0{\cdot}0034\,(4i^{\frac{1}{2}}+1)\,(T-T^\circ)$$

The observed change in cathode potential with current density is attributed to a hydrogen overvoltage which is controlled by the diffusion of hydrogen ions through a space-charged layer of nickel ions at the cathode surface. The equation does not apply at current densities below about 0·4 ma./cm^2.

The temperature coefficient of a nickel-plated electrode in the nickel-plating solution when no curretn flows is −0·0017 volt/°C.

The sudden change in slope of the E_c—T curves at about 0·42 volt and the hysteresis effect observed, are attributed to the adsorption of atomic hydrogen at the nickel cathode, the potential of −0·42 volt corresponding to an energy of desorption.

Nickel Plating of Aluminium-base Materials

'Plating on Aluminium.'
Steel, 1953, vol. 132, Feb. 16, pp. 76-8.

This article is based on a paper by W. G. ZELLEY (of Aluminum Research Laboratories), read before a recent meeting of the Electrochemical Society. It recommends the sodium-zincate pre-treatment, as being the most practical and economical method for plating aluminium and aluminium alloys. Optimum results, with regard to the corrosion-resisting properties of the coated parts, are obtained by ensuring good coverage by the thinnest possible zinc film.

Most of the zincate-dip baths now in use are within the following composition limits: caustic soda 400-500 g./L., zinc oxide 80-100 g./L. Research work at the Alcoa laboratories has shown that coverage, on all types of aluminium alloys, is improved by addition of a small amount of ferric chloride to such baths, plus some tartrate to maintain the iron in solution under the strongly alkaline conditions obtaining. With most of the alloys, coating with this type of film also improves the corrosion-resistance of the finally plated product. The modified zincate-dip process is beneficial also in plating aluminium alloys containing magnesium, where the double-dip method is sometimes employed (the first coating being removed by nitric acid prior to the second immersion). A preferred composition for the modified zincate bath is given as (g./L.): caustic soda 500, zinc oxide 20, ferric chloride 1, Rochelle salt 10. It is noted that a large excess of ferric chloride is to be avoided, since it tends to cause blistering of the deposits, and that when plating alloys of high magnesium content it is advisable to increase the Rochelle salt to 100 g./L. The modified solution is prepared by dissolving zinc oxide in the caustic solution in the usual manner, and adding the iron as a water solution of ferric chloride and Rochelle salt.

The very highly concentrated nature of the zincate baths renders them extremely viscous, which, in production operations, gives rise to problems of drag-out, and rinsing difficulties. The Aluminum Company is

investigating development of more dilute, less viscous baths, and it is already possible in some cases to use more dilute solutions if suitable additions are made to adjust the potential difference between zinc and the aluminium surface. Such modification keeps down the weight of zincate deposit, which tends to increase appreciably on dilution of the bath. Sodium nitrate (0·5-1·0 g./L.), and increase in Rochelle salt content are recommended for this purpose. Composition of a typical dilute bath is given as (g./L.): caustic soda 120, zinc oxide 20, Rochelle salt 50, ferric chloride 2, sodium nitrate 1. When dilute solutions are employed special attention must be given to prior preparation of the surface, and best results are obtained by a double zincate treatment, limiting immersion to 30 seconds, at a temperature not above 75°F. (24°C.). The Enthone Alumon process is a proprietary method based on the use of modified zincate solutions. The salts used in the Alumon bath remove oxide from the surface of the aluminium, activate it, and at the same time deposit a zinc-copper alloy which is adherent to the aluminium, and serves as a base for electroplating.

As an example of the improved corrosion-resistance obtained in aluminium-alloy parts plated after treatment by the modified zincate baths described, the author showed photographs illustrating the condition, after 300 hours' exposure to the salt-spray test, of panels nickel plated after preparation by (*a*) the regular, and (*b*) the modified, pre-treatment. Examples of good service with nickel+chromium-plated parts given include reference to bumpers of aluminium alloy, zincate-treated by the modified method, coated with a 0·0015 in. underlay of nickel, and finally chromium plated.

Determination of Total Sulphates in Nickel-Plating Baths

K. E. LANGFORD: 'The Determination of Total Sulphate in Nickel Plating Solutions, using Di-Sodium Ethylenediamine Tetra-Acetate.' *Electroplating*, 1953, vol. 6, Feb., pp. 41-2, 58.

As a further development of work on determination of magnesium in nickel-plating solutions described by the author in 1952 (*ibid.*, vol. 5, pp. 41-6; *Nickel Bulletin*, 1952, vol. 25, No. 4, pp. 99-100), the following method is recommended for estimation of total sulphates in nickel-plating baths:—

'Transfer a 1 ml. sample of the nickel solution to a 350 ml. Philips beaker and dilute to 100 ml. Raise to the boil and run in slowly from a burette 10 ml. N/5 $BaCl_2$-N/10 $MgCl_2$ solution. Mix and cool. Add 2 gm. NH_4Cl, 2 gm. KCN, 10 ml. NH_4OH and 6 spots of indicator.

'Titrate with N/5 E.D.T.A.* to the blue end point, allowing the precipitated $BaSO_4$ to settle for final confirmation. Let this volume of solution be X ml.

'*Blank*

Take 20 ml. (from a burette) of the combined N/5 $BaCl_2$-N/10 $MgCl_2$ solution and dilute to 100 ml. Add 2 gm. NH_4Cl, 2 gm. KCN, 10 ml. NH_4OH and 6 spots of indicator. Titrate with N/5 E.D.T.A. to the clear blue end point. Let this be Y ml. The figure need be checked only at very infrequent intervals.

$Y-X$= ml. of N/5 $BaCl_2$ equivalent to the SO_4

1 ml. N/5 $BaCl_2$ = 0·01421 gm. Na_2SO_4
= 0·02809 gm. $NiSO_4.7H_2O$
= 0·02629 gm. $NiSO_4.6H_2O$

20 ml. N/5 $BaCl_2$ will precipitate an amount of SO_4 equivalent to 45 oz./gal. Na_2SO_4 when using a 1 ml. sample of nickel solution. This quantity is adequate when dealing with most commercial baths.

Solutions

'*Combined $BaCl_2$-$MgCl_2$*

Weigh out 24·431 gm. 'Analar'-grade barium chloride ($BaCl_2.2H_2O$) and transfer to a 250 ml. beaker. Dissolve in the minimum quantity of distilled water. Weigh out approximately 2 gm. 'Analar' magnesium oxide (MgO) and dissolve in the minimum quantity of dilute hydrochloric acid (1:1). Transfer both solutions to the same 1 litre standard flask and make up to the mark with distilled water. Standardize as described under 'blank'. The $BaCl_2$ will be exactly N/5, but the titration is necessary to ascertain the strength of the $MgCl_2$.

'*N/5 E.D.T.A.*

Dissolve 38 gm. powdered disodium ethylenediamine tetra-acetate in distilled water and make up to 1 litre in a standard flask. Standardize against 'Analar' magnesium sulphate $MgSO_4.7H_2O$, taking sufficient salt to give a reading of about 20 ml. (0·5 gm.). Titrate after the addition of 2 gm. NH_4Cl, 2 gm. KCN, 10 ml. conc. NH_4OH and 6 spots of indicator.

'*Indicator*

0·5 gm. Erichrome Black T (Solochrome Black WDFA by I.C.I., Ltd.) and 4·5 gm. hydroxylamine hydrochloride are dissolved in 100 ml. of alcohol or industrial spirit.'

NON-FERROUS ALLOYS

Nickel-containing Coinage: Report of Royal Mint

'Eighty-First Annual Report of the Deputy Master and Comptroller of the Royal Mint, 1950.'
Published by His Majesty's Stationery Office, 1953, 90 pp. Price 3/6.

The Report contains the usual type of statistical data on coins struck by the Royal Mint and other Mints within the Commonwealth. Returns given include figures relating to U.K. silver and cupro-nickel currency, U.K. nickel-bronze currency, and details of coinage of all types struck in 1950. In the report of the Operative Department it is noted that successful results are being obtained with high-frequency furnaces which have recently been installed. Figures showing percentage of cupro-nickel bars passed for U.K. coinage in the years 1946 (97·28 per cent.), 1947, 1948, 1949 and 1950 (99·52 per cent.)

* Ethylenediamine Tetra-Acetate.

show that difficulties encountered when cupro-nickel was first used have been satisfactorily overcome. The trial of the Pyx, 1951, covered assay of cupro-nickel coins of 1950 production, made for New Zealand and for Southern Rhodesia.

Conservation of Nickel: Modified Regulations relating to Nickel Silver

See abstract on p. 50.

Metals Specifications Handbook

See abstract on p. 50.

Monel Stays in Standard Locomotives

'Standard Class '2' Locomotives for British Railways.' *Engineer*, 1953, vol. 195, Feb. 6, p. 207.

Description of the first of the new 2-6-0 standard class '2' mixed-traffic tender engines for British Railways, recently completed at the Darlington works. It is one of ten locomotives which will be placed in service on the Western Region, for duties similar to those of the '2251' and '2301' classes of the 0-6-0 classes of similar power. Although the new locomotive is the smallest tender engine to be built in the range of twelve for British Railways, it nevertheless incorporates, wherever suitable, modern developments which have been found successful in design of larger units. In this, as in earlier standard locomotives built under the current B.R. programme, firebox water space stays are of Monel, whilst roof longitudinal and transverse stays are of steel.

Nickel-Titanium Alloys: Constitution

H. MARGOLIN, E. ENCE and J. P. NIELSEN: 'Titanium-Nickel Phase Diagram.' *Jnl. of Metals*, 1953, vol. 5, Feb., Sect. 2, Trans. Suppl. pp. 243-7.

The paper reports investigation of the titanium-nickel phase diagram (*a*) up to 68 per cent. nickel, using alloys of iodide titanium and high-purity nickel and employing metallographic, melting point and X-ray methods of examination, and (*b*) up to 90 per cent. nickel by examination of as-cast structures of sponge-titanium-base alloys. The authors summarize their conclusions as follows:—

'(1) Eutectic decomposition of β containing 6-7 per cent. nickel, into α (less than 0·2 per cent. nickel) and Ti_3Ni (38 per cent. nickel), at 770°± 5°C.

'(2) Eutectic solidification of liquid containing 28-29 per cent. nickel, into β (13 per cent. nickel) and Ti_2Ni at 955°± 5°C.

'(3) Peritectic formation of Ti_2Ni from liquid (about 37·5 per cent. nickel) and TiNi (about 53 per cent. nickel) at approximately 1015°C.

'(4) Eutectic solidification of liquid (about 65 per cent. nickel) into TiNi (about 59 per cent. nickel) and $TiNi_3$ (78·6 per cent. nickel), at approximately 1110°C.

'(5) Eutectic solidification of liquid, to form $TiNi_3+\gamma$.

'The β phase may transform by a nucleation-and-growth process, or by a martensitic reaction. The transformation of β may be stimulated in the presence of α or Ti_2Ni, or may be suppressed on water quenching alloys containing 8 per cent. or more of nickel.'

Welding of 90-10 Cupro-Nickel Alloys

L. H. HAWTHORNE: 'Inert Arc Welding of 90-10 Cupro-Nickel.' *Welding Jnl.*, 1953, vol. 32, Jan., pp. 31-6.

Extensive tests, by the British Non-Ferrous Metals Research Association in England, and by Revere Copper and Brass Company in U.S.A., have shown that a 90-10 copper-nickel alloy containing about 1·5 per cent. of iron is a promising substitute for 70-30 cupro-nickel in some applications. The alloy has been approved by the U.S. Navy, and has been incorporated in specifications covering tube, bars, plates, rods, sheet and strip, for use, where suitable, in the place of the higher-nickel material. The (U.S.) National Production Authority has also required its use whenever possible.

This report deals with welding experiments on $\frac{1}{8}$, $\frac{1}{4}$, $\frac{1}{2}$ and $\frac{3}{4}$-in. plate of an alloy of the following composition: copper 88·49, iron 1·20, manganese 0·13, silicon 0·00, lead 0·008, zinc 0·03, aluminium 0·001, magnesium 0·05, sulphur 0·006, per cent., nickel remainder. The filler wire used was of essentially the same composition as the plate material, except that some tests were made with a higher manganese content (0·39 per cent.). The composition finally developed for the wire, which gave consistently satisfactory results, was copper 87·58, iron 1·54, manganese 0·17, silicon 0·12, titanium 0·29, lead 0·002, zinc 0·00, aluminium less than 0·001, magnesium 0·00, sulphur 0·002, per cent., balance nickel. Argon was used exclusively as the shielding gas.

It was originally intended that the investigation should be limited to gas-shielded metal-arc welding, but it was later decided to make some welds using the tungsten-arc method. The initial series of gas-shielded metal-arc welds was unsatisfactory, due to porosity at or near the fusion line. This could be eliminated by addition of titanium to the weld, a modification which was found beneficial in both series of tests.

The experiments showed that, using the filler alloy mentioned above, it is possible, by either of the methods used, to produce welds having good mechanical properties. Typical values are cited for welds made in plate of the various thicknesses, using filler rods of varying composition. It was found that the addition of silicon to the filler alloy containing titanium materially increases the tensile strength of the resulting weld. The alloy of the composition recommended appears to be immune from hot shortness.

Electrolytic 'Thinning' of Metals and Alloys

See abstract on p. 50.

Estimation of Silicon in Nickel Alloys

C. L. LUKE: 'Photometric Determination of Silicon in Ferrous, Ferromagnetic, Nickel, and Copper Alloys. A Molybdenum Blue Method.' *Analytical Chemistry*, 1953, vol. 25, Jan., pp. 148-51.

Preliminary qualitative experiments made by the present author, considered in conjunction with recent literature on photometric determination of silicon, indicated that it was likely to be impossible to develop a method applicable to a wide range of alloys

without resorting to separations prior to the photometric determination. The sodium diethyldithiocarbamate-chloroform extraction method used by Serfass *et al.*, for removal of nickel from nickel-plating baths prior to estimation of aluminium, proved to be satisfactory for removal of the metals which interfere in determination of silicon.

The method described by the author consists of solution of the sample in a mixture of hydrochloric and nitric acids, destruction of the nitric acid by heating with formic acid, removal of all interfering metals by a carbamate-chloroform extraction, and finally, determination of silicon by the usual photometric molybdenum-blue method, using, essentially, technique described by MINSTER (*Analyst*, 1946, vol. 71, p. 428; 1948, vol. 73, p. 507). Data confirming the accuracy of the method include estimations on standard ferrous and non-ferrous alloys of many types, among which are austenitic chromium-nickel steels, nickel-iron alloys, nickel-chromium alloys, and Monel. Results on three samples of standard nickel are also given. The method is applicable in the range 0·01 to 2 per cent. of silicon if the rather poor precision and accuracy in the upper ranges, which result from the limitations of photometric technique, can be tolerated.

NICKEL-IRON ALLOYS

Low-Expansion Nickel-Iron Alloys: Properties and Treatment

'Low-Expansion Nickel Alloys.'
Materials and Methods, 1953, vol. 37, Jan., p. 123.

Tabular data sheet, giving physical and mechanical properties, recommendations on treatment and fabrication, and main uses, for three groups of nickel-iron alloy, containing, respectively, 36, 42 and 47-50 per cent. nickel, balance iron.

Metals Specifications Handbook

See abstract on p. 50.

Electrolytic 'Thinning' of Metals and Alloys

See abstract on p. 50.

Determination of Sulphur in Nickel-Iron Alloys

K. SWOBODA: 'Determination of Sulphur in High-Nickel Steels.' *Radex Rundschau*, 1952, No. 7, pp. 299-302.

The author reports comparison of four methods for estimation of sulphur in an alloy containing nickel 80, molybdenum 4, per cent., iron remainder. The procedures examined were:—

(1) The evolution process (unsuitable for this type of alloy, since such material is not soluble in hydrochloric acid, the first step involved).
(2) The ether process.
(3) The reduction process.
(4) A process developed by the author, which is studied in considerable detail. This procedure involves combustion in a stream of oxygen, with addition of tin. The method is recommended as suitable in cases where a high degree of accuracy is not required, or as a rapid check of sulphur content. If a higher degree of accuracy is essential the reduction method, with subsequent iodometric titration, must be used. When using the reduction method for a series of determinations it is advisable, after completion of each estimation, to drive the hydrogen out of the reaction chamber and displace it with an inert gas, in order to have the apparatus ready for the next sulphur determination.

CONSTRUCTIONAL STEELS

Line Broadening of Martensite in Nickel Steels

R. A. SMITH: 'Line Broadening of Martensite in Nickel Steels.' *Jnl. Iron and Steel Inst.*, 1953, vol. 173, Feb., pp. 147-52.

The broadening of X-ray diffraction-line profiles is, in general, attributed to two types of lattice imperfection: (1) differences in parameter due to the presence of residual internal strains, and (2) a small particle-size effect, which implies the formation of some type of substructure within the original grains. The broadening expected from these two forms of imperfection has been calculated: some details of equations derived by earlier investigators are given in the introduction to this paper.

In the work now reported 3 per cent. nickel steels of medium (0·28-0·35%) and low (0·10-0·15%) carbon content were used, instead of the plain-carbon types employed in previous investigations of line broadening. It is pointed out that this modification has advantages, in that the martensite in the nickel steels has a body-centred cubic crystal structure, and that the small amount of retained austenite in low-nickel steels quenched to room temperature has a negligible effect on the shape of the lines. In consequence, the diffraction pattern obtained with the nickel steels is much less complicated than that given by carbon steels, and it is not necessary to use the elaborate technique which has been employed in earlier work, to resolve the individual martensite lines.

The X-ray diffraction line shapes were recorded in a Geiger counter spectrometer. Fourier analysis was used to determine the true physical broadening, from the observed line shapes of the martensite and of annealed iron, which is assumed to exhibit only experimental broadening. Further information on the nature of the transformed material was obtained from a consideration of the Fourier coefficients.

The results indicate that the effects produced by martensitic transformation are similar to those found in cold-worked metals: the unusually large broadening in martensite is not a consequence of a separate phenomenon. The major part of the line broadening may be attributed to distortion of the lattice during transformation, but the observations made suggest

that a substructure, which can cause some broadening, is formed. Carbon content is effective in determining the degree of internal strain remaining after transformation.

Thermal Stabilization of Austenite in Iron-Carbon-Nickel Alloys

E. R. MORGAN and T. KO: 'Thermal Stabilization of Austenite in Iron-Carbon-Nickel Alloys.'
Acta Metallurgica, 1953, vol. 1, Jan., pp. 36-48.

It has been known for many years that the progress of the austenite→martensite transformation can be influenced by rate of cooling, and it has also been observed that if the cooling through the martensite range is interrupted and the steel is held isothermally for a certain period, transformation does not necessarily resume immediately when cooling is continued, but only after a certain temperature lag, which may result in a loss of martensite when the steel is quenched to a fixed reference temperature. This phenomenon is known as 'stabilization of austenite', but the authors are of the opinion that the term 'thermal stabilization' is to be preferred, in order to differentiate between this effect and the retarding influence of plastic deformation, on the martensite transformation, which may be designated 'mechanical stabilization'.

Degree of thermal stabilization can be expressed in two ways:—(i) in terms of the increase, δ, in the amount of retained austenite when the steel is quenched to a reference temperature, and (ii) in terms of the difference, θ, between the temperature at which cooling is interrupted, and that T_s, at which the martensitic transformation begins again when cooling is resumed. Extent of stabilization has been found, in several types of steel, to increase with time, and rate of stabilization has been observed to increase with rising temperature. The effect of temperature on degree of stabilization in a given steel is, however, influenced by the amount of prior transformation which the steel has undergone.

Following a brief survey of earlier investigations on thermal stabilization in nickel steels, the authors report observations on a series of iron-carbon-nickel alloys containing 0·9-1·3 per cent. carbon and 0-10 per cent. nickel. All the materials except a commercial carbon steel (included for comparison purposes) were melted under argon, using electrolytic iron, carbonyl nickel and carbon. Only a small percentage of impurities was present: a typical analysis is given as manganese 0·04, silicon 0·07, sulphur 0·020, phosphorus 0·020, per cent.

Details are given of austenitizing and quenching procedure, and of the method used for estimation of amount of martensite in the alloys. The experimental results are reported in the form of transformation curves for the respective materials, and a report of observations on thermal stabilization occurring (1) above M_S and (2) below M_S. The all-over results are critically considered, in the light of theories of stabilization which have been advanced by various investigators: (*a*) stress-relaxation theory, (*b*) composition change theory, (*c*) exhaustion of nuclei theory.

The conclusions are that for steels containing about 1 per cent. of carbon and 0-5 per cent. of nickel:—

'(i) Stabilization of austenite occurs during isothermal holding above and below M_s. The rate of stabilization varies with the composition of the austenite, increases with increasing temperature, and is greatly increased by the presence of martensite.

'(ii) The stabilization above M_s causes a depression of M_s and increases the amount of austenite at a reference temperature near M_s.

'(iii) The difference in the amounts of retained austenite in a steel quenched at various rates is chiefly due to stabilization above M_s during quenching.

'(iv) The amount of martensite lost due to athermal and isothermal stabilization can be recovered by quenching to a sufficiently low temperature.'

It is suggested that stabilization is caused by an increase in the resistance of austenite to martensite formation, and that this increase is mainly due to the formation of Cottrell atmospheres round dislocations.

Machinability Tests on Cast and Wrought Nickel-Alloy Steels

'Machinability Studies Compare Cast and Wrought Steels.' *Iron Age*, 1953, vol. 171, Jan. 22, pp. 102-4.

This article is based on an investigation carried out by the Steel Founders' Company of America. Seven types of cast steel, widely used in industry, were prepared, in 18 different conditions; their machining characteristics were studied, and some comparisons were made with wrought steels of similar compositions. The materials used included nickel-chromium-molybdenum steels of the S.A.E. 4340, 8630 and 8640 types, and chromium-molybdenum 4130 and 4140 steels. Tool life was used as a criterion of machinability, both carbide and high-speed tools being used.

The conclusion drawn from these tests, which are considered to be of a preliminary nature, is that when high-speed steel tools are used cast and wrought steels having similar microstructures have comparable machining characteristics. When carbide tools are used the machinability of the cast and wrought steels is generally comparable, but at a speed of 300 f.p.m. the behaviour of the wrought steels is better, indicating that they are less abrasive. At higher speeds abrasiveness is not so important a factor in tool breakdown as the temperature developed at the tool/steel interface, and at speeds above 450 f.p.m. the cast steels had better machining qualities than wrought steels of comparable types.

Physical Constants of Steels at Elevated Temperatures

BRIT. IRON AND STEEL RESEARCH ASSOCN. (editor): 'Physical Constants of Some Commercial Steels at Elevated Temperatures.' Foreword by DR. EZER GRIFFITHS. *Published by Butterworths Scientific Publications*, London, 1953: 38 pp. Price 21/-.

In the years preceding World War II the National Physical Laboratory undertook the determination of the physical constants of a series of typical steels selected for their commercial interest. The work was carried out for the Thermal Treatment Sub-Committee

of B.I.S.R.A. (formerly a committee of the Iron and Steel Institute). The results of these determinations have, from time to time, been published in the form of scientific papers. The data obtained in the whole series of investigations at the N.P.L. have now been tabulated in a form from which the constants for a particular steel at a given temperature can easily be obtained. These constants are of interest to engineers, metallurgists, and others concerned with the heat-treatment of steels.

The determinations made by the National Physical Laboratory covered measurement of specific and total heats between 50° and 1200°C., coefficients of thermal expansion (between 0° and t°C.) up to 1100°C., electrical resistivities between 0° and 1300°C., and thermal conductivities between 0° and 350°C. The data so obtained have been arranged in tabular reference form, involving some interpolation and extrapolation of data, which has been carried out by the Physics Department of the British Iron and Steel Research Association. In addition to the constants obtained by direct measurement, the following have been derived by calculation: mean coefficients of thermal expansion between t° and (t+50)°C.; densities at elevated temperatures; thermal diffusivities. Total heat-temperature graphs and expansion-temperature graphs are given for ranges of temperature covering the transformations: from these curves it is possible to obtain some indication of the heats of transformation, and the volume changes which occur during transformation.

Other information contained in the volume includes properties of super-cooled austenite, at temperatures down to 200°C. below the A_3 temperatures, determinations of electrical resistivity, and thermal conductivity of Armco iron and of some recently developed high-temperature steels.

Attention is directed to data on isothermal transformations and complete S curves of a series of En steels, published in Iron and Steel Institute Special Report No. 40. Many of these steels are similar in composition to those referred to in the present publication.

Influence of Hydrogen on Mechanical Properties of High-Alloy Nickel-containing Steels

See abstract on p. 64.

Metals Specifications Handbook

See abstract on p. 50.

Spectrographic Analysis of Low-Alloy Steels

BRIT. IRON AND STEEL RESEARCH ASSOCN.: 'Spectrographic Analysis of Low-Alloy Steels.'
Iron and Steel Inst. Special Report No. 47; 83 pp. Published by the Institute, Sept. 1952; Price 15/- (to Members of the Institute 7/6.)

Variations in spectrographic technique employed in different laboratories for the analysis of iron and steel have been examined by the Spectrographic Analysis Sub-Committee of the British Iron and Steel Research Association, and, based on the most successful of the alternative procedures examined, a recommended method for spectrographic analysis of low-alloy steels has been drawn up. This procedure has been given extensive trials on a series of chemically analyzed standards, and satisfactory results have been obtained. Details of the method are published in this report, together with a summary of information used in formulating it. It is applicable to analysis of low-alloy steels having an iron content of 95%±1%: for compositions outside this range a correction is necessary. The elements which may be determined, and the concentration ranges covered, are as follows: silicon 0·05-0·80, manganese 0·05-1·50, nickel 0·10-5·00, chromium 0·05-3·00, molybdenum 0·05-1·50, vanadium 0·03-0·65, copper 0·05-0·50, per cent.

The applicability of alternative procedures and accessory equipment has, in several instances, been described, and, where necessary, theoretical considerations and detailed operating instructions have been included. The text is divided into sections corresponding broadly to the sequence of analytical operations required, and references to the literature have been included to facilitate more detailed study of the subject. The *bibliography* contains over 150 references.

HEAT- AND CORROSION-RESISTING ALLOYS

High-Temperature Materials: Typical Compositions and Properties

J. B. MEIERDIRKS: 'Simplification and Some Potentialities of High-Strength Heat-Resisting Alloys.'
Amer. Iron and Steel Inst., Preprint of paper presented at Philadelphia Regional Technical Meeting, Dec., 1952; 30 pp.

This is essentially a review type of paper. The various factors affecting strength at high temperatures are considered, and the basic principles which have been applied in developing materials having satisfactory high-temperature properties are discussed. The alloying elements introduced to enhance the properties of heat-resisting materials are enumerated, and their functions are explained in relation to the following effects which they exert:—

(1) Direct strengthening of the material by virtue of their presence in the solid solution.
(2) Causing the formation of ageing precipitates, and determining the conditions of their occurrence.
(3) Increasing the resistance to grain rotation required for creep, by formation of ageing or non-ageing precipitates at the grain boundaries.
(4) Changing the cold-working characteristics in hot-cold work, and reducing the rate of relaxation of this cold work at service temperatures.
(5) Controlling grain size through formation of precipitates, or by directly affecting grain growth and nucleation.

The various types of high-temperature material are considered on the basis of examples of representative

materials (mostly of U.S. production) in each class, as shown below:—

A. *CAST AND WROUGHT ALLOYS, TYPE I*

Examples: X-40 and S-816.

Service temperatures: 1450°-1550°F. (785°-840°C.).

Treatment: wrought alloys usually solution treated and aged: cast alloys age during service at elevated temperature.

Production: cast alloys and some wrought alloys are induction melted.

Composition:—

(1) Total of about 32 per cent. chromium, molybdenum, tungsten and niobium, chromium being 20 per cent. minimum, to provide oxidation-resistance. Use of tungsten predominates in the strongest alloys of this class.
(2) Balance iron, nickel, cobalt, with cobalt predominating, and iron at a minimum in the strongest alloys of the class.
(3) Carbon 0·4-0·5 per cent. in cast alloys; variable in wrought alloys. In high-carbon wrought alloys niobium is added as stabilizer; in other wrought alloys carbon is low and niobium is absent.
(4) Experimental cast alloys; not yet fully tested. Characterized by high nickel, no cobalt, low iron content, and relatively large amounts of special elements (boron, beryllium, vanadium, titanium, aluminium).

B. *WROUGHT ALLOYS, TYPE II*

Examples: Waspaloy; M-252.

Service temperatures: 1400°-1500°F. (760°-815°C.).

Treatment: solution treatment and ageing.

Production: induction: arc melting not yet successfully developed.

Composition:—

(1) Average of 24 per cent. chromium, molybdenum, titanium and aluminium, but such total content is variable. Slightly less than 20 per cent. chromium; titanium 2·5 and aluminium 1, per cent., as major strengtheners; molybdenum up to 10 per cent. in some alloys, for additional strength.
(2) Balance, iron, nickel, cobalt, with nickel predominating: the strongest alloys of the class contain 10 or 20 per cent. cobalt; iron is low in the strongest alloys, but up to 20 per cent. in others.
(3) Carbon is low in all the alloys of this group (0·05-0·15 per cent.).

C. *INTERMEDIATE ALLOYS*

Examples: A-286, 16-25-6.

Service temperatures: 1200°-1350°F. (650°-735°C.).

Treatment: most of these alloys are hot-cold worked, to give high-temperature strength. A-286 and Discaloy are solution treated and aged.

Production: electric arc melting, in medium-sized furnaces.

Composition:—

(1) Average of 20 per cent. chromium, molybdenum, tungsten, etc. Chromium averages about 15 per cent.; other additions vary, but average about 4 per cent. Titanium is used to produce ageing in A-286 and Discaloy.
(2) Balance iron, nickel and cobalt, with iron predominating. Nickel 9-26 per cent., averaging 20 per cent., to provide stability; G 18B is the only cobalt-containing alloy of this group.
(3) Carbon varies, but is usually low: in G 18B niobium is used to stabilize, in view of the higher carbon.

A series of charts illustrates range and typical compositions of the respective groups, as represented by a wide variety of materials made by U.S. producers. Tables show nominal compositions of typical heat-resisting alloys, with some limiting stress-rupture values. In view of the difficulty sometimes experienced in finding nominal compositions for some of the less well-known proprietary alloys, a list is given below of those for which information is given in this paper:

Cast Alloys: Alloy 61 (H.S.23), Vitallium (H.S.21), X-63, X-40 (H.S.31), L-251, 422-19 (H.S.30), 6059 (H.S.27), Refractaloy 80, H.E. 2048, Hastelloy C, I-1360, 95-M-255.

Wrought Alloys, Type I: L-605, I-336, W.F.31, S-844 (V.36), S-816, 25 Ni, Refractaloy 70, S-590, N-155, Hastelloy X, C.S.A. 39, Hastelloy B.

Wrought Alloys, Type II: K 42B, Refractaloy 26, Nimonic 90, Waspaloy M-252, Nimonic 80A, Inconel X.

Intermediate Alloys: G. 18B, 16-25-6, Discaloy, A-286, 17W, H.S. 88, 19-9DL.

Other Jet-Engine Alloys: Inconel, A.I.S.I. Types 310 and 321, Crucible 422, H-40, 17-22 A.S.

The author makes some comments on applications other than aircraft gas turbines for which materials of the respective types are likely to be used.

Metals Specifications Handbook

See abstract on p. 50.

Creep Characteristics of Nickel

T. H. HAZLETT and E. R. PARKER: 'Nature of the Creep Curve.' *Jnl. of Metals*, 1953, vol. 5, Feb., Sect. 2, Trans. Suppl., pp. 318-23.

The work reported in this paper was carried out to determine the effects of variable factors such as stress, temperature, alloying, and structure, on the creep characteristics of metals. Nickel was selected as the basis material for examination: the analysis of the specially prepared metal used is given as (% by weight): iron 0·030, silicon 0·003, calcium 0·001, magnesium 0·020, sulphur 0·002, per cent., nickel (by difference) 99·94 per cent. Details of the purification process employed in preparing the material for test are to be given in a forthcoming paper. The authors discuss at some length the progress of creep at various stages, and the accuracy of various modes of presentation of creep data. Curves relating to nickel-base alloys (nickel-titanium and nickel-tungsten), aluminium, lead, and zinc are included, in support of various aspects of the discussion.

From correlation of the data obtained in the authors' own experiments with results of other investigators, it is concluded that the creep rate for structurally stable metals tested under conditions of constant stress

decreases continuously until the initiation of failure: there is no region of constant creep rate. It is considered that the creep curve may be accurately represented by the empirical equation

$$\varepsilon - \varepsilon_o = At^b$$

at constant temperature, or

$$\varepsilon - \varepsilon_o = Bt^b e^{-Q/RT}$$

over a considerable range of temperature.

'The parameter ε_o is a quantity having real physical significance, denoting an instantaneous strain upon loading. The parameter A varies in a regular manner with stress and alloy content and exponentially with $1/T$. The parameter b also varies over a wide range, but the correlation of b with temperature, stress, grain size, and alloy content will require many additional tests for complete elucidation.

'The empirical equation given is applicable to both single crystals and polycrystalline materials, indicating that the continuously decreasing creep rate is not solely a grain-boundary phenomenon.

'Accurate determination of the parameters A, b and ε_o requires strain measurements beginning at very short times after applying the load. In many cases readings must begin only a few seconds after loading.' Tests of this type were made by the authors, using specially constructed high-speed strain-recording equipment.

Influence of Boron on Oxidation-Resistance of Chromium-Nickel-Cobalt-Iron Alloys

W. O. BINDER and E. D. WEISERT: 'Some Notes on the Oxidation-Resistance of Boron-containing Chromium-Nickel-Cobalt-Iron Alloys.' *Corrosion*, 1953, vol. 9, Feb., N.A.C.E. Conference Section, pp. 28, 30.

Abstract of paper to be given at Ninth Annual Conference of National Association of Corrosion Engineers, 1953.

The authors report experiments in which a series of alloys containing 18 and 20 per cent. of chromium, with various percentages of nickel, cobalt, iron, and boron, were subjected to oxidation tests in air at 1650°, 1830° and 2012°F. (900°, 995° and 1100°C.). Temperatures somewhat above those encountered in gas-turbine operation were used in order to accelerate the attack, since the effect of boron was not readily noticeable below 900°C.

The results demonstrate the beneficial influence of chromium and nickel on oxidation-resistance, but show that iron and boron, and, to a lesser extent, cobalt, have an adverse effect. Molybdenum was highly deleterious in this respect. Carbon and tungsten, at the levels studied (not stated in the abstract), had no apparent effect.

The authors conclude that while boron has an adverse influence on the oxidation-resistance of the alloys investigated, it may be possible, by correct control of composition, to produce boron-containing alloys which have oxidation-resisting qualities comparable to those of boron-free types. This will probably be accomplished primarily by keeping the iron content below 6 per cent., and by the use of tungsten rather than molybdenum as a minor addition for strengthening the alloys.

Thermal-Shock Tests on Austenitic and Ferritic Materials

W. C. STEWART and W. G. SCHREITZ: 'Thermal Shock and Other Comparison Tests of Austenitic and Ferritic Steels for Main Steam Piping. A Summary Report.' *Amer. Soc. Mechanical Engineers, Paper* 52-A-35, Nov.-Dec., 1952; 15 pp. plus tables and figures.

The superior mechanical properties of austenitic chromium-nickel steels at temperatures above 1000°F. (540°C.) has aroused much interest in connexion with main steam lines of naval vessels. This superiority has been recognized in the A.S.M.E. Boiler Code, in that higher working stresses are permitted for austenitic than for ferritic steels. There has, however, been but little experience with austenitic steels in main steam lines, and reports of failures, in the oil industry, of pipes conveying hot oil from cracking stills have raised some doubt as to the suitability of austenitic materials for steam service. Although ferritic steel pipes have been known to develop cracks as a result of thermal shock due to periodic flow of hot oil, their susceptibility to this form of deterioration is less than that of the austenitic types. In view of the possibility of deterioration from thermal shock occasioned by carry over of boiler water into steam lines, and of the need for further knowledge of the properties of austenitic welds joining austenitic pipe to castings, and of joints between austenitic and ferritic materials, extensive tests have been carried out at the U.S. Naval Engineering Experiment Station, Annapolis, by the authors of this paper and their colleagues. A previous paper recorded results of thermal-shock and other comparative tests on austenitic and ferritic steel piping for high-temperature steam service (*Trans. Amer. Soc. Mech. Eng.*, 1950, vol. 72, pp. 1043-55; *Nickel Bulletin*, 1950, vol. 23, No. 12, p. 234). Since that time tests of full-scale thermal-shock specimens and mock ups, as well as supplementary laboratory-scale tests, have been completed. The present paper records the new test data and summarizes the results of the whole of the investigation to date.

Thermal-shock tests of 6-in. pipe and valve assemblies in both 18-8-Nb and 2¼Cr-1 Mo steels are described. The specimens used were of two weights, schedule 80, corresponding to 900 p.s.i. working pressure, and schedule 160, corresponding to 2,000 p.s.i. working pressure, at 1050°F. The 160-schedule specimens each contained a section of dissimilar pipe which introduced transition and composite welded joints. The shock treatment was designed to simulate the quenching action which might result from the carry over of boiler water into a pipe line carrying steam at 1050°F. Each assembly was subjected to 100 shocks.

The effect of mechanical loading, on full-scale members, was investigated by testing mock-ups designed to simulate expansion bends, and two mock-ups were subjected to a range of reversed bending stress to failure. The condition of the welds, as affected by the thermal-shock treatment, and the mock-up test, is discussed. The report also contains a considerable amount of high-temperature data obtained on laboratory specimens, taken from pipe and casting assemblies,

which were fabricated and heat-treated in the same manner as the full-scale test members. These include tensile properties at temperatures up to 1100°F. (595°C.) and stress-rupture, creep, and fatigue properties at 1000° and 1100°F. (540° and 595°C.).

Reactions between Refractory Oxides and Metals at High Temperatures

G. ECONOMOS: 'Behaviour of Refractory Oxides in Contact with Metals at High Temperatures.' *Industrial and Engineering Chemistry*, 1953, vol. 45, Feb., pp. 458-9.

Modern high-temperature requirements in connexion with nuclear-energy developments and supersonic flight necessitate, in some cases, use of metals in contact with refractory oxides. The qualitative investigations reported in this paper were made (at Massachusetts Institute of Technology) as a preliminary to more extensive research on combinations which might find useful application. The refractory oxides selected for the experiments were of commercially available types, but of high purity: the Al_2O_3, MgO, BeO, TiO_2 and ThO_2, were 99+%, and the ZrO_2, of the same degree of purity, was stabilized with about 4 per cent. of CaO and contained about 4 per cent. HfO_2. The oxides were fabricated into discs, and sintered, and the surface to be placed in contact with the metals was given a high polish, to minimize purely mechanical adherence during test. In addition, TiO_2 was reheated in vacuo to 1600°C., giving it a black colour. The metals titanium, beryllium, silicon, niobium, nickel, molybdenum and zirconium (either pellet or powder of 99+% purity) were placed on the flat oxide discs, and over each assembly was placed a recessed cover disc of the appropriate oxide. The composites were fired in an inert atmosphere, in an induction furnace, at 1400°, 1600° and 1800°C. Each firing contained assemblies comprising one oxide with all the metals. Experimental test conditions are described and illustrated.

After cooling, under controlled and uniform conditions, all samples were examined to ascertain physical changes such as corrosion, oxidation, adherence, volatilization, and discoloration. Metallographic and petrographic examination was used to determine alterations of the oxide phases, and the nature of the reaction products formed was examined by X-ray analysis.

The results led to the following conclusions:—

At temperatures below 1400°C. the oxides Al_2O_3, ZrO_2, BeO, MgO, TiO_2 and ThO_2 were essentially unaffected by the metals beryllium, zirconium, titanium, niobium, molybdenum, nickel and silicon. At 1600° and 1800°C. the relative order of decreasing stability of the oxides was found to be ThO_2, ZrO_2, Al_2O_3, BeO, MgO, TiO_2. Molybdenum and nickel were the only metals of those tested which were stable in contact with the oxides at all temperatures. Beryllium and silicon showed early reactivity with some of the oxides, and attacked all the oxides at the more elevated temperatures used. Zirconium and titanium, which have high oxygen solubility, reacted with the oxides, due, it is believed, to some form of oxygen-exchange mechanism. Niobium showed reactivity mostly at the more elevated temperatures. This and a few other reactions could be explained as solid solutions at these temperatures, but further investigation is required to obtain more conclusive proof on this point.

Ceramic Coating of Heat- and Corrosion-Resisting Materials

D. K. KROSCH: 'High Temperature Ceramic Coatings in Industrial Applications.' *Materials and Methods*, 1953, vol. 37, Jan., pp. 162, 164, 166, 168.

The article is based on data assembled by the Solar Aircraft Company, on the behaviour of ceramic-coated austenitic steels and high-temperature alloys, in furnace fixtures and other parts used in high-temperature operations, and in chemical-engineering equipment subject to corrosive conditions. Typical examples of increased service life obtained in coated parts are quoted.

Creep Testing of Nimonic Alloys

C. W. WEAVER: 'Specification Creep Testing of Nimonic Gas-Turbine Alloys.' *Bull. Inst. Metals*, 1952, vol. 1, Feb., pp. 168-72.

This paper, contributed to a symposium on new techniques of testing, describes (with illustrations) machines and technique developed in the laboratories of The Mond Nickel Company, Limited, to meet the growing demand for short-time creep tests such as those required by D.T.D. 725, 736 and 747, on materials for gas-turbine rotor blading and other high-temperature applications. Compactness and simplicity of design, and robustness, are outstanding features. Specifications for Nimonic 80A and Nimonic 90 call for tests of 75 hours' duration, at 17 and 19 tons per square inch, respectively, in both cases at 750°C. Tests of long duration, at temperatures up to 900°C. are required on experimental alloys.

Subsurface Porosity Caused by High-Temperature Corrosion

A. DE S. BRASUNAS: 'Subsurface Porosity Developed in Sound Metal during High-Temperature Corrosion.' *Metal Progress*, 1952, vol. 62, Dec., pp. 88-90.

The author records observation of subsurface voids, in Inconel and other high-temperature alloys, associated with processes involving depletion of chromium as a result of (1) leaching with certain molten salts, (2) high-temperature vacuum treatment, and (3) high-temperature oxidation.

(1) *Fused alkali metal fluoride salts* in static contact with chromium-containing alloys such as Inconel, at temperatures above 1300°F. (705°C.), resulted in porosity, several mils deep, in the surface layer. The attack was accompanied by a drop in the chromium content of the metal affected, from about 15 to about 5 per cent., and analysis of the fluoride bath used as the corroding medium showed correspondingly higher chromium-to-iron and chromium-to-nickel ratios than would be obtained by uniform attack of the alloy, thereby clearly establishing the selective removal of

chromium which had occurred. Saturation of the fluoride salt with chromium, prior to test, prevented this formation of voids in tests which without such treatment would have produced voids several mils deep.

To prove that the voids were the result of chromium depletion, two other methods of test were used: results are briefly summarized below:—

(2) *High-temperature vacuum treatment.* Specimens of Inconel and of 80-20 nickel-chromium alloy were exposed to a vacuum of 0·1 mm. Hg for 42 hours at 1375°C. (2500°F.) and were furnace cooled. Chemical analysis of the Inconel sample indicated a drop in chromium content from 14 to 8 per cent. Both specimens showed many subsurface voids, those in Inconel being spherical, those in the 80-20 alloy angular.

(3) *High-temperature oxidation.* The resistance of chromium-containing alloys is attributable to formation of a chromium-rich and diffusion-resistant oxide phase on the surface of the alloy, and since the chromium in the oxide phase is supplied by the surface and subsurface regions of the specimen, chromium depletion of these layers necessarily results. An Inconel specimen exposed for 200 hours to air at 1250°C. (2280°F.) showed the usual layer of oxide on the surface, and oxide penetration of the adjacent grain boundaries for a short distance. In addition, much deeper subsurface voids were observed.

The tests reported all confirm that voids observed under the conditions described are caused by depletion of chromium. As chromium atoms are taken away from the surface, by one or other of the means considered, a concentration (activity) gradient results, which causes chromium atoms from the underlying region to diffuse towards the surface, leaving behind a zone enriched with vacancies. The amounts tolerable increase with rise of temperature. Diffusion is visualized as taking place principally by movement of vacancies, i.e., neighbouring atoms move to occupy vacant sites. In reactions in which the net effect of the diffusion involved is essentially mono-directional, there is a movement of mass which results in a change in density and/or shape of that portion of the specimen. If the concentration of vacancies left behind is greater than some critical value, they will tend to collect at suitable locations, and appear as visible voids. The mechanism of subsurface void formation, showing movement of vacancies, is diagrammatically illustrated in this note, in relation to alloys containing nickel, chromium and iron.

Contamination of Condensate in Heat Exchangers: Tests on Nickel-containing Alloys

J. D. RISTROPH and E. B. POWELL: 'Contamination of Condensate by Heat Exchanger Tube Alloys.' *Amer. Soc. Mechanical Engineers, Paper* 52-A-63, Nov.-Dec., 1952; 12 pp., plus tables and figures.

The accumulation of foreign metal and metallic oxides on the internal heating surfaces of high-pressure boilers has become a source of increasing concern in the steam-power industry in the U.S.A., and during the past ten years, with the advent of the 2,000 p.s.i. and higher-pressure steam generators, such deposits have been found to constitute serious hazards, retarding heat transfer, and so tending to develop high temperature in the tube walls, resulting in overheating and/or corrosion. Deposits of copper origin appear to have the further undesirable property of masking the evolution of hydrogen from the affected surface, thus preventing advance warning of impending failure. Operating experience has indicated corrosion and solution of metals of the steam-condensate feed-water part of the system, external to the boiler proper, as the major source of the metallic deposits in 100%-condensate-feed-water type of plant. It is to this form of plant that the present paper refers.

The authors report results of an investigation of heat-exchanger-tube alloys carried out on behalf of the Virginia Electric and Power Company, in connexion with plans for erection of three additional 750,000 lb. steam per hour, 1,555 p.s.i. gauge controlled-circulation boilers. The programme was planned to use test equipment which would employ steam condensate as produced in actual power plants, and to simulate conditions of normal operation. Additional tests were arranged to study the influence of certain special chemicals which might effect reduction in corrosion and solution of piping and other parts of the steam-condensate system. Detailed notes are given on design and operation of the test apparatus.

The following tube alloys were selected for test, as affording reasonable coverage of commercial materials appropriate for feed-water heat-exchanger tubing: phosphorized copper, 88-10-2 copper-zinc-tin; antimonial, arsenical, and phosphorized Admiralty metal; aluminium bronze; arsenical aluminium brass; 90-10 copper-nickel alloys containing 0·8 per cent. iron; 70-30 copper-nickel alloys containing 0·4 or 0·6 per cent. iron; Monel; Type 316 (molybdenum-containing) chromium-nickel stainless steel.

Results of Tests

Under conditions average for all the tests, all the materials used develop protective films which tend to lessen their contaminating effects. Under the conditions used in these tests, normal for the respective power plants under consideration, most of the twelve materials tested would initially give relatively high rates of condensate contamination, but within a few hundred operating hours the rate of contamination from any one of the alloys would attain a maximum, and would then progressively decrease to a relatively low rate, peculiar to the individual material and the chemical and physical environments involved. On the basis of the tests described, Monel 70-30 cupro-nickel and arsenical Admiralty metal would be grouped as heat-exchanger alloys of non-ferrous or copper-bearing type alloys showing least tendency to contribute copper contamination to the condensate. Aluminium bronze and aluminium brass tend to contribute most highly to copper contamination. Under conditions of feed-water treatment normal for the plants on which the tests were made, Type 316 stainless steel appeared to contribute iron contamination

at rates not much below those for copper contamination shown by non-ferrous alloys in corresponding test runs. Increase in temperature of the condensate caused increased contamination from all the materials tested, but with many of them the influence of differences in gas concentration outweighed the effects of substantial variations in temperature.

Treatment of feed water with cyclohexylamine effected marked decrease in rates of contamination imparted by all the non-ferrous alloys, and entirely prevented iron contamination from the stainless steel. The response of the nickel-bearing non-ferrous alloys to the cyclohexylamine treatment was less marked than that of the other non-ferrous alloys; in feed water so treated aluminium bronze, phosphorized copper and arsenical Admiralty metal occupied the most favourable positions.

Rate of condensate contamination to be expected from contact with any given material is strongly influenced by the nature and extent of prior non-metallic contamination of the condensate, and possibly also by prior metallic contamination, and by the arrangement and dimensions of the metal surfaces giving contact exposure to the condensate path. Continuance and intimacy of contact are determining factors.

Resistance of Cast Iron-Chromium-Nickel Alloys to Corrosion by Flue Gases

J. H. JACKSON, C. J. SLUNDER, O. E. HARDER and J. T. GOW: 'Resistance of Cast Iron-Chromium-Nickel Alloys to Corrosion in Oxidizing and Reducing Flue-Gas Atmospheres.' *Amer. Soc. Mechanical Engineers, Paper* 52-A-37, Nov.-Dec., 1952; 16 pp. plus figures.

This paper is one of a series reporting research at Battelle Memorial Institute, sponsored by the Alloy Casting Institute, the broad aim of which is to determine the behaviour of cast alloys in various conditions of high-temperature service, and to assess the relative suitability of the different materials for specific applications. Some of the major results obtained in other parts of the research, and data published by other investigators, are summarized in the introduction to the present paper, which covers tests in which iron-chromium-nickel alloy castings were exposed, at 1800°-2000°F. (980°-1095°C.) to typical sulphur-bearing flue gases, representing both oxidizing and reducing conditions. Details of the synthetic atmospheres are given.

The alloys used ranged in composition from 11 to 36 per cent. chromium and from 0 to 70 per cent. nickel, and were representative of commercial heat-resisting alloys used as castings. Details are given in the Table, which also shows the A.C.I. designation for the respective alloys. (For convenience, this nomenclature is used in the abstract.) Most of the test exposures were of 100 hours' duration: some tests were prolonged to 1000 hours, and the effects of cyclic-temperature variations, and of alternating exposure to oxidizing and reducing atmospheres, were also examined.

As a supplement to tests on alloys of the types shown in this table, an examination was made of the corrosion-resistance of alloys of some of these basic types to which substantial additions of aluminium, silicon, vanadium, or molybdenum had been made.

A.C.I. Designation	Cr %	Ni %	C %	Si %	Mn %
HE	26-30	8-11	0·2-0·5	2 max.	2 max.
HF	18-23	8-12	0·2-0·4	2 max.	2 max.
HH	24-28	11-14	0·2-0·5	2 max.	2 max.
HK	24-28	18-22	0·2-0·6	3 max.	2 max.
HT	13-17	33-37	0·35-0·75	2·5 max.	2 max.
HU	17-21	37-41	0·35-0·75	2·5 max.	2 max.
HW	10-14	58-62	0·35-0·75	2·5 max.	2 max.
HX	15-19	64-68	0·35-0·75	2·5 max.	2 max.

Behaviour was evaluated in terms of metal loss (which is an indication of the amount of surface metal consumed through scaling), and in terms of the total depth of metal affected by the corrosive attack (which includes not only the metal completely consumed by scaling, but also the depth of metal below the scale-to-metal interface affected by the attack). This latter type of corrosion consists in decarburization, intergranular penetration, and massive infiltration.* All three types of deterioration are illustrated. Much detailed information is given in the report with regard to experimental conditions, and the resistance of the respective alloys to the various forms of attack mentioned above. From the observations made, the following conclusions are drawn:—

Corrosion of cast iron-chromium-nickel alloys in oxidizing and reducing flue-gas atmospheres is a complex phenomenon, on which many factors of composition, design, and operating conditions have a pronounced influence. It is recommended that engineers designing with such materials should base their selection rather on data indicating resistance to subsurface attack than on rates of surface scaling and metal loss alone.

On the basis of the 100-hour tests it was found that under reducing conditions rate of attack increased as the sulphur content of the gas rose from 0 to 500 gS†. Increase in chromium content of the alloys was beneficial in lowering the rate of attack. In atmospheres containing less than 100 gS increase in nickel content also effected some improvement, but in higher-sulphur atmospheres nickel contents above about 15 per cent. were associated with increased corrosion. Under oxidizing conditions the effect of sulphur up

*This term is used to describe areas where the corrosion product had penetrated the metal to such a degree that only isolated islands of metal remained.

†gS = grains of sulphur per 100 cubic feet of gas. In an oxidizing flue gas 1 gS would be equivalent to 0·0016% SO_2 by volume: in a reducing atmosphere it would be equivalent to 0·0016% H_2S. 1 gS = 0·023 gram S/m³.

to about 100 gS was similar to that observed under reducing conditions, but in this case the amount of chromium required to secure good resistance to corrosion was less as the nickel content was increased. In the higher-sulphur atmospheres attack was much less severe in the oxidizing than in the reducing conditions.

The effect of cyclic fluctuations in temperature appeared to be dependent on the other test conditions. In reducing atmospheres cycling caused considerably more metal loss in alloys containing over about 10 per cent. of nickel, but from the standpoint of total depth of metal affected cyclic fluctuations had relatively little influence in alloys in the 10-40 per cent. nickel range. Slightly greater corrosion was caused by cycling in alloys containing less than 10 per cent. of nickel. In oxidizing atmospheres the differences in corrosive attack caused by cyclic-temperature variation were relatively small.

Raising the temperature from 1800° to 2000°F. (980° to 1095°C.) resulted in more severe attack: in reducing atmospheres containing 500 gS at 2000°F. (1095°C.) rates of attack were very high even in nickel-free 30 per cent. chromium alloy.

As the time of exposure was increased from 100 to 1000 hours there was a marked reduction in rate of attack in both reducing and oxidizing flue-gas atmospheres containing 100 gS. Acceleration of attack with prolonged exposure therefore appears to be unlikely under such conditions.

The HE-, HK- and HH-type alloys showed the best general corrosion-resistance under the various test conditions. The HW type was generally better than the HT: both showed good performance until the sulphur content of the gas was raised to 100 grains per cu. ft. The HX and HU types were generally better than the HW and HT types, but they too were inferior to the HE, HK and HH types when the sulphur contents were of the order of 100 grains per 100 cu. ft. or above. The HF type, originally designed for use at temperatures below 1600°F. (870°C.), gave reasonably good performance, and at 1800°F. (980°C.) in reducing atmospheres containing at least 100 grains of sulphur per 100 cu. ft. this type of alloy was slightly superior to the HW and HT types.

Although in the higher-sulphur atmospheres the high-chromium low-nickel alloys HE, HD and HC are more resistant to corrosion than the higher-nickel austenitic alloys, the lower mechanical strength of the former at elevated temperatures is a balancing factor which should receive consideration in design. Although cycling of temperature was, in general, detrimental, cycling of the type of atmosphere, from reducing to oxidizing, did not appear to increase rates of corrosion.

Additions of silicon (1·5-2·5 per cent.) resulted in marked reduction of corrosion: many commercial alloys contain about 1-2 per cent. of this element. Aluminium (about 1 per cent.) was also beneficial, but additions of molybdenum and vanadium resulted in slightly increased rates of attack.

It is emphasized that the data reported in this paper should not be applied to wrought heat-resisting alloys, due to the lower carbon contents of the latter and their greater instability.

Welding of High-Temperature Materials for Gas-Turbine Parts

K. H. KOOPMAN: 'Shielded Arc-Welding Processes for Jet-Engine Components.' *Welding Jnl.*, 1953, vol. 32, Feb., pp. 103-15.

The author presents a well-arranged discussion of the spheres of application, in welding of jet-engine materials, of the inert-gas-shielded tungsten arc, inert-gas-shielded metal arc, and submerged-arc processes. The subject is dealt with from the point of view of the essential characteristics and operation of the processes, their advantages, apparatus required, and typical applications. The paper is well illustrated.

Following the descriptions of the respective processes and recommendations for their use, a table is given showing the principal alloys and high-alloy steels used in jet engines, with details of nominal compositions, S.A.E.(A.M.S.) specification numbers, jet-engine components for which the various alloys are used, and recommendations on choice of welding rod. The materials included in the table are classified in three groups, covering, respectively, wrought alloys which are mainly modified austenitic stainless steels; wrought specialized alloys of nickel- and/or cobalt-base, and iron-containing types in which the iron is less than 50 per cent.; cast special alloys. The tabular information is supplemented by notes on features peculiar to individual alloys within the series, which require consideration in connexion with welding. A further table lists various combinations of dissimilar alloys which may have to be joined in assembly of jet-engine parts. Recommendations are made on selection of welding rods for joining the dissimilar materials.

X-Ray Fluorescent Analysis of High-Temperature Alloys

R. M. BRISSEY: 'Analysis of High-Temperature Alloys by X-Ray Fluorescence.' *Analytical Chemistry*, 1953, vol. 25, Jan., pp. 190-2.

In view of the complexity of high-temperature alloys, methods of analysis which do not involve separation of the material into its component elements are particularly attractive. One method of this type which is now gaining popularity is X-ray fluorescent analysis, in which the alloy is subjected to X-radiation of sufficient energy to excite the characteristic radiation of all the elements in the sample, the intensity of the characteristic lines being a function of the concentration of the respective constituents. Instruments capable of producing, resolving, and measuring the intensity of characteristic radiation are now on the market: the unit described in this paper is a General Electric Company model. Mode of operation of the analyzer is described, and a chart shows a recording made with it of characteristic X-ray lines from a typical high-temperature alloy. Some comparison of values obtained by X-ray fluorescent and standard chemical-analysis methods is given. It is considered that the new technique has a promising future for routine control determinations of major constituents in alloys. Attention is, however, drawn to two definite limitations: (1) radiation from elements having atomic

numbers below 22 is highly absorbed by air, the diffracting crystal, and the counter window, and for this reason these lighter elements are not easily determined by X-ray fluorescence; (2) the lower limit of concentration which can reliably be determined depends on the intensity of the characteristic line measured with respect to the background intensity, which, in turn, depends on the element and the matrix in which it occurs. In general, concentrations less than 1 per cent. by weight may not be determined satisfactorily in a short counting time.

Influence of Hydrogen on Mechanical Properties of High-Alloy Nickel-containing Steels

J. D. HOBSON and J. HEWITT: 'The Effect of Hydrogen on the Tensile Properties of Steel.' *Jnl. Iron and Steel Inst.*, 1953, vol. 173, Feb., pp. 131-40.

Earlier papers in this series described the effects of hydrogen on the tensile properties of low-alloy steels, and the distribution of hydrogen in large masses of steel (*Jnl. Iron and Steel Inst.*, 1951, vol. 169, pp. 209-20; 1952, vol. 170, pp. 118-22: *Nickel Bulletin*, 1952, vol. 25, Nos. 1 and 4, pp. 15 and 107). The present paper contains a report of additional tests on low-alloy steels (including the influence of variables such as speed and temperature of tensile testing), and also gives results of tests on some austenitic steels. The high-alloy types studied included a 26 per cent. nickel steel, a 14½ per cent. manganese steel and five varieties of austenitic nickel-chromium steel.

The main conclusion of the earlier studies was that the previous heat-treatment of the sample was very important in determining the degree of hydrogen embrittlement. Provided that this factor was taken into account, ductility, as measured by percentage reduction in the tensile test, was found to decrease in a regular manner with increasing hydrogen content. In the case of forged steel, the direction of the flow structure in the specimen must also be considered.

In the investigation described in the present paper the high-pressure heating hydrogenation technique (*loc. cit.*) was again employed, and hydrogen was determined by vacuum heating.

If the discontinuity-migration theory of hydrogen embrittlement, which involves movement of atomic hydrogen by diffusion to discontinuities in the crystal structure during deformation, is accepted, it is assumed that molecular hydrogen accumulates in the microcavities and, by building up a pressure, leads to triaxial stresses, causing failure at external stresses lower than those which would cause failure in the absence of hydrogen. Assuming this theory to be correct, it should be possible to avoid embrittlement resulting from the presence of hydrogen by testing either at high speeds or at low temperatures, since either condition would minimize the deleterious influence of migration of hydrogen. Experimental results reported in this paper confirm that hydrogen embrittlement decreases with increasing speed of loading, in tension, bend, or Izod tests. In torsion tests (made on a 3 per cent. chromium-molybdenum steel only) no effect of hydrogen was observed, either at slow or faster rates of test. Results of fatigue tests appeared to indicate that hydrogen definitely lowers the number of stress reversals necessary to cause failure.

For determination of the influence of the temperature of test on hydrogen embrittlement effects the tests were made on the 3 per cent. chromium-molybdenum steel: properties were measured on hydrogenated specimens at 20°, 100°, −78° and −196°C. The results, in accordance with theoretical expectation, lead to the general conclusion that hydrogen is less deleterious at low temperatures than at room temperature. It was also observed that the stress (and presumably also the microstructure) exhibiting minimum sensitivity to hydrogen is a function of temperature; a detailed study is made of the nature of the low-temperature fractures in this steel. Further tests on the same steel showed that when treated to very high tensile strength it is very susceptible to hydrogen embrittlement at room temperature and that at −196°C. it becomes very notch-sensitive, a characteristic which is intensified when it contains small amounts of hydrogen.

In the tests on the austenitic steels the materials were used in the fully softened condition. In most cases, hydrogen could be introduced into these steels by the high-pressure technique, provided that a long soaking time was given, to allow for the much slower diffusion of hydrogen in austenite than in ferrite. Precautions required with some of the steels to prevent austenite breakdown during hydrogenation are noted. The solubility of hydrogen in austenite is some 3-4 times its value in ferrite at a given temperature, and it is therefore possible to introduce reasonable quantities either at low temperatures and high pressure, or at high temperatures and atmospheric pressure. All the possible methods were employed in introducing hydrogen into the steels of the austenitic group. All were found to be relatively immune to the influence of hydrogen: in many cases large amounts, up to 25 ml. per 100 g., were needed to produce a measurable effect on ductility, as gauged by reduction in area in the tensile test. Since the steels examined were of widely different composition, it would appear that their tolerance to hydrogen does not depend on the presence of a particular alloying element, but that it is rather an intrinsic property of the austenite crystal lattice. Tests on two austenitic chromium-nickel steels showed that cold-drawing renders these steels more susceptible to embrittlement by hydrogen than are the fully softened steels of similar composition, but in this condition the effect is still only relatively slight as compared with the influence of hydrogen in ferritic steels. Tests on the hydrogenated austenitic steels at various temperatures showed that ductility is significantly affected only in the case of test pieces broken at −78°C.

It is emphasized that the results presented in this and the earlier papers make it apparent that hydrogen embrittlement is not so erratic a phenomenon as it is usually thought to be, provided that all the significant and related variables are controlled. It is obvious that hydrogen is not the only factor responsible for formation of hairline cracks, but that it needs to be associated with transformational or cooling stresses, as has been urged by some other investigators.

Chi Phase in Molybdenum-containing Stainless Steels

P. K. KOH: 'Occurrence of Chi Phase in Molybdenum-bearing Stainless Steels.' *Jnl. of Metals*, 1953. vol. 5, Feb., Sect. 2, Trans. Suppl., pp. 339-43.

Molybdenum additions, although desirable as a means of enhancing resistance of certain types of austenitic nickel-chromium steel to corrosion, and of increasing strength in high-temperature alloys, have been found to stimulate formation of sigma phase. The chi phase, which in certain steels co-exists with sigma, is closely similar to it in form (see *Metal Treatment*, 1951, vol. 18, pp. 300-11; *Nickel Bulletin*, 1951, vol. 24, Nos. 9-10, pp. 205-6).

The present paper gives results of some metallographic and X-ray diffraction studies on experimental chromium-molybdenum-iron alloys, chromium-nickel stainless steels, titanium- and tungsten-bearing high-chromium steel containing 1·62 per cent. molybdenum, and nickel- and cobalt-base alloys each containing chromium 25, molybdenum 10, per cent. The etchant used for delineation of chi and sigma was boiling 50 per cent. hydrochloric acid solution.

The report is illustrated by many photomicrographs, and details are given of the chemical analysis of isolated phases from the various specimens, diffraction data on the chi phase, and micro-hardness measurements on the chi phase formed under various conditions of heat-treatment.

The phase is found to be essentially an intermetallic compound of chromium, molybdenum, and iron. It resembles the sigma phase in its metastability, chemical composition, and brittleness. The close similarity in composition of chi and sigma in the 317 steel leads to the suggestion that chi may be an ordered structure of sigma, which is known to be of a distorted body-centred cubic structure.

Chi phase was found in as-cast 23-10 chromium-molybdenum-iron alloy, as well as in heat-treated molybdenum-containing chromium-nickel steels of the 316, 316L and 317 types, and in the modified high-chromium steel. No corresponding phase was found in either the nickel- or cobalt-base alloy containing chromium and molybdenum.

In austenitic stainless steels of the molybdenum-bearing types cold working appears to promote sigma formation, but to suppress formation of chi phase. In applications involving extended time at elevated temperature, formation of chi or sigma phase appears to depend on whether the previous history involved solution treatment or cold work. In welded sections, riveted joints, or other strained areas of large vessels, where solution treatment is not possible, sigma formation will, it is considered, be unavoidable, possibly without even partial formation of chi.

Structural Hardening of Austenitic-Ferritic Steels: Utilization of Sigma Phase

J. HOCHMANN: 'Structural Hardening of Stainless Austenitic-Ferritic Steels.' *Revue de Métallurgie*, 1952, vol. 49, Dec., pp. 849-54.

The author discusses the hardening of austenitic steels by (1) precipitation of carbides, (2) boron additions, and (3) the presence of intermetallic phases resulting from additions of aluminium+titanium, or beryllium. Treatments essential for hardening by these three methods are briefly reviewed, and attention is also directed to the marginal-austenitic steel of the 17-7 chromium-nickel type containing titanium+aluminium, in which a high degree of hardening can be produced by suitable heat-treatment.

The major part of the paper is devoted to a consideration of the structural hardening obtainable in steels of duplex structure (austenitic-ferritic): the experiments described were made on a steel of the following composition:—carbon 0·05, chromium 25, nickel 8, molybdenum 2·5, copper 1·5, silicon 0·5, manganese 0·5, per cent. In the aggregate of austenite and ferrite of which such a steel is composed, the phase susceptible to hardening is the ferrite, and the hardening effect is produced by heating in either of two ranges of temperature: (1) 400°-500°C., or (2) 600°-800°C. Treatment of straight-chromium steels in the lower of these ranges produces a material which is hard, but brittle, and is characterized by severely reduced resistance to corrosion: in consequence, this type of treatment has found no useful practical application in such steels. In high-chromium steels (chromium over 30 per cent.) the sigma phase formed by treatment in the 600°-800°C. range results in considerable hardening of the steel, but in this case also the associated brittleness precludes utilization of the method in service. The author's experiments on the austenitic-ferritic chromium-nickel steel are, however, designed to emphasize that, in the duplex-structure steel, tempering at 450°C. hardens the ferrite without producing the undesirable brittleness found in the straight-chromium steels, and that formation of sigma produced by heating at 800°C. results in a useful degree of hardening which, although associated with reduction in ductility, can be appropriately utilized for parts in which no elastic or dynamic stresses are involved, but in which high hardness and resistance to seizing are essential. With regard to corrosion-resistance, the author states that behaviour of such hardened steels is satisfactory in severely corrosive media, e.g., hot dilute sulphuric acid, cold dilute hydrochloric acid, and saline solutions. The 450°C. tempering treatment causes lowering of resistance to attack by boiling nitric acid, but in the sigma-hardened steel produced by treatment at 800°C. resistance to this corrodent remains good.

Influence of Cold Working on Magnetic Properties of Austenitic Chromium-Nickel Steels

S. STORCHHEIM: 'Permanent Magnetic Properties in 18-8 Obtained by Cold Working.' *Iron Age*, 1953, vol. 171, Feb. 12, pp. 140-2.

The author reports experiments on three grades of 18-8 steel, having the following compositions:—

Steel No.	Ni %	Cr %	Mn %	Si %	C %
1	8·30	18·64	0·55	0·54	0·15
2	8·70	18·49	0·44	0·46	0·19
3	10·67	19·15	1·04	0·51	0·076

The effect of varying percentages of cold work was studied on these materials, using wire of 0·028-in. diameter, which, after annealing in a dissociated-ammonia atmosphere at 2150°F. (1175°C.) was passed through a series of diamond dies of progressively smaller diameter, in a conventional wire-drawing unit. The wire was then drawn at 250 f.p.m. for several minutes, to ensure uniformity in rate of reduction. (Speed of reduction had been shown to have an effect on properties: see paper abstracted in *Nickel Bulletin*, 1953, vol. 26, No. 1, p. 24.) Wires of steels 1 and 2 were then given a 92 per cent. reduction, and wire of steel 3 (which, due to its higher nickel content, was less susceptible to transformation of austenite to martensite) was reduced by 98 per cent. Tests made on the steels, in the original and final conditions, included determinations of remanence and coercive force, and, in the case of steels 1 and 2, measurements of tensile strength and of electrical resistivity. Results are shown in a series of tables and graphs: some notes on the main observations made are given below.

Remanence increased in a similar, parabolic, manner for all three steels, as the percentage cold reduction was increased, but in steel 3, containing the higher amount of nickel, appearance of any appreciable amount of ferromagnetic constituent occurred only at about 50 per cent. reduction (whereas it was at 15 per cent. reduction for the other two steels). At 92 per cent. reduction wire of steel 2 showed a remanence value 12·9 per cent. greater than that of steel 1 and 597 times greater than that of steel 3, this latter large difference being attributable to the higher nickel of steel 3. The carbon content of the steels appeared to exert no appreciable influence on the remanence values. For coercive force, with increasing percentages of cold reduction a decreasing curvilinear relationship was found. As with remanence, initiation of measurable coercive force in steel 3 was at a higher degree of reduction than in the other two steels, and H_c values for the higher-nickel steel were markedly larger, for all percentage reductions of area, than those of steels 1 and 2. The influence of carbon was again indiscernible. Comparison of the H_c and B_r values developed at given percentages of reduction showed that as (with increasing cold reduction) the B_r increased, H_c decreased. Mechanical tests confirmed published data, showing that there is a continuous rise in tensile strength with increasing percentages of cold reduction: the greater carbon of steel 2 resulted in higher tensile values for a given degree of cold work than those found for steel 1. Although considerable scatter occurred, the points on the curve electrical resistivity vs. cold work indicate a general rise in resistivity with increasing percentages of reduction. For a given amount of cold reduction the steel of higher carbon content, No. 2, showed larger values of resistivity.

Conservation of Nickel: Modified Regulations Relating to Stainless Steels

See abstract on p. 50.

Physical Constants of Steels at Elevated Temperatures

See abstract on p. 56.

Stress-Corrosion Cracking of Chromium-Nickel Austenitic Steels

C. EDELANU: 'Transgranular Stress Corrosion in Chromium-Nickel Stainless Steels.' *Jnl. Iron and Steel Inst.*, 1953, vol. 173, Feb., pp. 140-6.

The paper is based on investigations made in the Brown-Firth Research Laboratories: the discussion is restricted to the transcrystalline type of stress-corrosion failure, and does not deal with intercrystalline corrosion, which has already been extensively studied.

The service failures examined in this laboratory have occurred in most cases in solutions containing chlorides, at elevated temperatures, and have also been found in material which had been in contact with impure steam. Similar failures have been reported as being reproduced in boiling concentrated chloride solutions, and in ethyl chloride. Magnesium chloride is most widely used as the testing medium, and all the usual grades of stainless steel crack when stressed in this type of solution.

Following a condensed review of earlier work on the factors affecting stress-corrosion, the author reports tests on the following types of steel: 18-8, low-carbon 18-10, 18-8-Ti, 18-8-Mo, 12-12 and 25-20 chromium-nickel steels, 18-18 chromium-nickel steel containing copper and molybdenum, 17-2 chromium-nickel steel, and 20, 17 and 13 per cent. straight-chromium steels. Full details of composition and of preparation of the specimens are given. The test pieces comprised 3-in. × ½-in. specimens cut from 18-gauge material, bent into a U. The U-shaped samples were forced into 18-8 chromium-nickel steel holders, so that an elastic stress was maintained. Glass slides on the side of the holder prevented electrical contact between the holder and the specimens. Specimens were bent by placing them on two rollers between which a plunger was forced so that a standard bend was maintained: the value of the stress in the bent specimens is not known, but must be regarded as appreciably above the yield point of the steels.

Tests made in boiling 42 per cent. magnesium chloride solution classified the steels into three main groups: (1) those based on the 18-8 composition, which are highly susceptible to stress corrosion in such conditions, (2) steels having higher nickel contents, which are less susceptible, but gave somewhat scattered results, and (3) ferritic and martensitic steels, which appear to be immune from deterioration in the test conditions used. (It is, however, noted that martensitic steels are subject to other types of stress corrosion.) A second series of tests, made on experimental steels in which the chromium and nickel contents were varied, broadly confirmed conclusions drawn from the tests on the commercial steels. Results on other specimens, based on the 18-8 composition, but containing copper, manganese and nitrogen in higher-than-normal amounts, indicated that these additions do not cause great change in stress-corrosion properties. Steels stabilized with titanium or niobium, and others containing molybdenum, showed the same general tendencies as those observed in the straight 18-8 steels.

Investigation of the influence of nature and concentration of the test solutions proved that many chlorides in addition to magnesium chloride can cause stress corrosion. Neutral types, such as sodium and potassium chloride, are dangerous, but concentrated zinc, calcium, and magnesium chloride appear to be even more so, due either to their much higher boiling points, or because of slight acidity. The progress of the cracking varied somewhat in the different types of solution. Influence of concentration of the test medium was determined on sodium- and magnesium-chloride solutions only: the results indicate that cracking can occur in solutions much less concentrated than those commonly used for testing susceptibility to stress corrosion. Tests were also made in sodium-chloride solutions to which other media had been added. These showed that oxidizing agents can greatly accelerate stress corrosion: addition of dichromate to a 3 per cent. sodium chloride solution produced particularly spectacular results. In concentrated magnesium-chloride solution dichromate additions were less effective, but the presence of ferric chloride caused considerable acceleration of cracking.

Examination of stress-cracking susceptibility in low-carbon 18-10 and titanium-stabilized 18-8 steels annealed at various temperatures shows that treatment at temperatures above 850°C. should reduce internal stresses to a value below that at which danger from stress corrosion is considerable.

Exposure of stressed specimens for 1300 hours in the steam space of a boiler operating at about 190°C. and 175 p.s.i. confirmed that stress-corrosion cracking can occur in an atmosphere of impure steam, even in a highly corrosion-resistant material such as the 18-18-Cu-Mo steel, which is relatively resistant to cracking in a magnesium-chloride solution.

Microscopical examination of the cracks in the steels, in relation to structure, supported the view that the transgranular stress corrosion of austenitic chromium-nickel steels is due to preferential attack on small quantities of quasi-martensite, which form in this type of steel when it is strained. The susceptibility can be reduced by increasing the nickel content (and thus increasing the stability of the austenite), or by reducing the alloy content (thus producing a steel which contains large quantities of quasi-martensite and in which corrosion is therefore more widely distributed). Increasing the chromium content also results in some improvement, presumably because the austenite in the two-phase steels produced by this means is relatively stable at low temperatures.

Corrosion-Resisting Materials in Chemical Plant of Celanese Corporation of America

D. R. KECK: 'The Dependence of the Canadian Chemical Company on Mining and Metallurgy.'
Canad. Mining and Metallurgical Bull., 1953, vol. 46, Jan., pp. 28-31.

This paper describes some of the problems associated with the processing units of the petrochemical plant operating, under the Canadian Chemical Company, Limited, at Bishop, Texas, and discusses features of plant of a somewhat similar character being erected at Edmonton for production and processing of cellulose acetate flake and organic chemicals. The aim of the paper is to show the dependence of the operations described on the use of suitable materials of construction. Some of the specific problems recorded as having been encountered in the early days of the Bishop plant are noted below:—

Corrosion, in fractionating towers, of wartime Type 316 austenitic steel containing excessively low molybdenum. Permission has been obtained to use steel of the following composition: carbon 0·07 max., molybdenum 2·25 min., chromium 17 min., nickel 11-14, per cent. This has proved satisfactory.

Intergranular corrosion due to faulty welding of Type 316 steel. Welding procedure involving use of low amperage and avoidance of exposure of the metal to prolonged heating during welding has now solved this problem.

Stress cracking of stainless-steel tubes, due to the high chloride content of the cooling water. This has been remedied by use of water of lower chloride content; substitution, in some parts, of Admiralty metal for stainless steel tubing, and other modifications of operating conditions and chemical compositions of liquid streams in contact with equipment.

Other problems briefly referred to as having been studied in the course of large-scale production in the Celanese Corporation plant include crevice corrosion, pitting of gasketted joints in stainless-steel piping (due to use of unsuitable gasket materials), concentration-cell corrosion in underground water piping, and fretting corrosion.

Soil-Corrosion Tests on Irons and Steels

I. A. DENISON and M. ROMANOFF: 'Corrosion of Low-Alloy Irons and Steels in Soils.'
Jnl. of Research, Nat. Bur. Standards, 1952, vol. 49, Nov., pp. 315-24; R.P. 2367.

In 1932 the National Bureau of Standards, in co-operation with manufacturers and users of piping, initiated an extensive investigation of selected materials employed in underground construction. Examination of test specimens removed after relatively short periods of exposure indicated that certain alloy steels showed corrosion-resistance superior to that of ordinary irons and steels. Specimens representing a wider variety of alloy irons and steels were therefore installed at intervals at the chosen test sites.

The condition of materials removed after successively longer periods of exposure has been described in a series of reports, and in 1950 a final report on specimens buried in 1932 was published (*ibid.*, 1950, vol. 44, p. 47).

The present report contains the results of measurements of weight-loss and pitting, made on 10 varieties of iron and steel, buried in 1937 and removed from the respective sites after five different periods of exposure, ranging from two to thirteen years. (For details of soils at test sites, installation procedure, and methods first used for cleaning the specimens, see the 1950 report referred to above, also *N.B.S. Circular* 450, published in 1945. Specimens subsequently removed were cleaned by immersion in fused sodium hydride.)

The materials covered in the present report are of the

following types: open-hearth steel; copper-molybdenum open-hearth iron; copper-nickel steel (0·95/0·52 per cent.); nickel-copper steel (1·96/1·01 per cent.); chromium-silicon-copper-phosphorus steel; 2 per cent. chromium steel containing 0·57 per cent. molybdenum; and 4-6 per cent. chromium steel with or without a small amount of molybdenum.

The corrosion of the individual materials in the various conditions of exposure is illustrated by an extensive series of photographs, and loss-in-weight data are tabulated in relation to the respective soils. The observations recorded show that the general effect of the alloying elements in the steels and irons tested was to reduce the initial rate of corrosion as measured by loss in weight, but that initial rate of pitting was increased. Except in very poorly aerated soils, however, rate of pitting of the alloy steels diminished more rapidly with time than in plain steel, with the result that the maximum depths of pits after the longest periods of exposure were less on the alloy steels than on the plain steels. Chromium effected the most pronounced reduction in weight-loss, but to secure maximum reduction in pitting the presence of molybdenum was also necessary. The chromium+molybdenum combination was also particularly effective in reducing corrosion of steel by cinders.

Pickling of Austenitic Chromium-Nickel Steels

G. BATTA, L. SCHEEPERS, L. WINANDY and G. DALLEMAGNE: 'Pickling of Special (Stainless) Steels.' *Revue de Métallurgie*, 1953, vol. 50, Jan., pp. 49-56.

The authors report laboratory investigations on the nature of the oxide film formed on 18-8 stainless steels, and the relative advantages of various types of pickling solution. The steel used contained carbon 0·04, silicon 0·41, sulphur 0·006, phosphorus 0·012, manganese 0·48, nickel 8·35, chromium 18·39, per cent., molybdenum trace. Oxidation was effected by repeated heatings, for periods of 30 minutes, at 1100°C. The oxide detached after the first cycle of heating and cooling contained only iron; after the second heating traces of chromium were present, and this constituent increased progressively with number of heatings. Only after six heating and cooling cycles was nickel found in the oxide layer. After repeating the cycle ten times the oxide detached from the specimen showed the following composition: Fe_2O_3 92·5; NiO 3·5; Cr_2O_3 3·9, per cent.

Three series of pickling experiments were carried out, to determine optimum baths, concentrations, etc., for satisfactory and economic removal of oxide, with minimum loss of metal. The first series covered the use of (*a*) hydrofluoric/nitric, and hydrochloric/nitric baths, (*b*) baths containing hydrofluoric, hydrochloric, and nitric acids, and (*c*) acid baths containing various inhibitors (of which only urea appeared to have any useful influence). In the second series a study was made of the action of hydrofluoric acid solutions containing varying amounts of ferric sulphate, while the third series covered investigation of replacement of hydrofluoric acid by less expensive acids. In this third group baths of the following types were used: hydrochloric acid/ferric sulphate, sulphuric acid/ferric sulphate, nitric acid/ferric sulphate, and hydrochloric- and sulphuric-acid-base baths containing various amounts of nitric acid, as used for pickling of steels of ordinary types. Metal loss, surface condition obtained, and the sensitivity of the pickling results to the initial surface condition of the steels, are considered.

On the basis of the detailed results reported, the authors urge the practical interest of the use of ferric sulphate as an oxidizing agent in hydrofluoric-acid solutions, or, as providing less expensive baths, solutions of sulphuric- or hydrochloric-acid base containing ferric-sulphate additions. Such solutions lessen the metal loss associated with pickling in hydrochloric-hydrofluoric-nitric acid mixtures, and will, it is believed, make possible the use of inhibitors the action of which is nullified in the ternary acid baths. It is suggested that the laboratory results should be further tested under semi-industrial conditions.

Copper-Sulphate/Sulphuric-Acid Test for Stainless Steels

L. H. SATZ: 'Utility of the Strauss Test.' *Metal Progress*, 1953, vol. 63, Jan., pp. 136, 138.

In this note the writer critically summarizes the main literature on the copper-sulphate/sulphuric-acid test, on which the amount of data is surprisingly small in view of the widespread use of the test for detection of susceptibility to intergranular corrosion. The efficiency of this type of solution depends on the passivating effect of the concentration of $CuSO_4$: the data which the writer quotes illustrate the differences in behaviour resulting from variations in concentration of the solution.

The conclusion is reached that 'since testing periods longer than 72 hours are finding increasing use for Strauss test sensitivity detection in laboratory investigations, the point as to whether sufficient passivity is obtained with $CuSO_4.5H_2O$ contents less than approximately 100 g./L. becomes important. The experimental answer to this question may hasten the time for adoption of one Strauss test solution which will fill most of the needs of industry and the research laboratory, and so lead to standardization, with all its advantages.'

PATENTS

Separation of Nickel and Cobalt from Acid Solutions

Solutions obtained by acid leaching of ores containing nickel and cobalt together with iron, copper, and other metals are treated (after removal of such other metals by known procedures) by the following process:—The solution is concentrated with ammonium salt of the acid corresponding to the anions in the solution, and there are simultaneously added sufficient ammonium ions to produce a pH of 4·5-6·7. The precipitated nickel-bearing crystals resulting from this treatment are collected. There is then added to the remaining cobalt-bearing solution sufficient ammonia to produce a pH of at least 6·8, and the

resultant solution is finally treated with a non-sulphide, sulphur-free reducing gas (preferably carbon monoxide or hydrogen) until precipitation of metallic cobalt is complete.

CHEMICAL CONSTRUCTION CORPN., assignee of E. S. ROBERTS, P. J. McGAULEY and F. A. SHAUFELBERGER. *Brit. Pat.* 688,574.

Removal of Nickel from Solutions containing Cobalt and Nickel

Nickel is precipitated from solutions containing both cobalt and nickel by means of metallic cobalt with at least one element of the group sulphur, arsenic, antimony, selenium, tellurium, in elemental or oxide form. Part of the cobalt may be combined with part or the whole of the other elements when in elemental form.

STÉ. GÉNÉRALE MÉTALLURGIQUE DE HOBOKEN. *Brit. Pat.* 683,679.

Treatment of Nickel Oxide Prior to Refining to Metallic Nickel

A material consisting essentially of oxides of one or more of the metals nickel, cobalt, copper is rendered more suitable for refining to metal by treating it, in the form of particles or small pieces, with a hydrocarbon gas which flows between the particles while they are moving downwards at reducing temperatures, thus effecting partial reduction of the material and simultaneously depositing finely divided carbon through the material. The product is cooled in a non-oxidizing atmosphere, to a temperature below that at which re-oxidation by air would take place. Suitable apparatus for carrying out the process is described.

MOND NICKEL CO., LTD. *Brit. Pat.* 686,921.

Recovery of Nickel from Solutions

Nickel is recovered from dilute solutions, e.g., mine waters, ore washings, and spent electrolytes, by precipitation with an oxime. The nickel-oxime compound is separated off and treated with an acid (e.g., sulphuric acid), so that the nickel dissolves and the oxime remains and can be re-used. Dimethylglyoxime is the preferred oxime.

MOND NICKEL CO., LTD. *Brit. Pat.* 686, 221.

Nickel Salts as Stabilizers in Polythenes

The patent relates to polythenes containing a minor proportion (preferably 0·5-5·0 per cent.) of a nickel salt of a dialkyldithiocarbamic acid having the formula $RR'NCS_2H$. (R and R′ are alkyl radicals containing together at least 8 carbon atoms) The presence of such a salt minimizes or prevents deterioration of the polythene on exposure to light. Nickel dibutyldithiocarbamate is the preferred salt.

IMPERIAL CHEMICAL INDUSTRIES, LTD. *Brit. Pat.* 684,976.

Production of Porous Sintered Metal Parts

The patent relates to porous shaped metal articles suitable in particular as electrodes for alkaline accumulators, which are made by sintering, preferably in a hydrogen atmosphere. The metal powder used as the initial material is produced by alloying a metal powder of low bulk density by contact with the salt of a more electro-positive metal. In an example carbonyl-nickel powder of bulk density 0·6 kg./L. is heated for an hour at 50°C. in a copper-nitrate solution, charged while still moist into a sintering mould, and heated in a stream of hydrogen, first at 500°C. and then at 1000°C., to form a nickel-copper plate having a pore volume of 85 per cent.

BADISCHE ANILIN- UND SODA-FABRIK. *Brit. Pat.* 686,148.

Porous Nickel Plates for Alkaline Accumulators

A highly porous accumulator electrode is made by sintering a layer of metal powder (e.g., iron or nickel) preferably of density less than 1·5 g./cm³ and filling the pores with the active material. The powder is preferably backed by a metal foil or gauze carrier to which the metal powder is applied as a suspension in a liquid, after which the treated product is given a reducing sintering treatment at 600°-1000°C. The plate is not more than 1 mm. thick.

H. VOGT. *Brit. Pat.* 687,569 (corresponding to Swiss Pat. 279,737 and French Pat. 985,685).

Sintered Nickel Accumulator Plates

Positive electrodes of alkaline accumulators consist either of a porous sintered plate of nickel, iron, cobalt, or an alloy or mixture of these metals, having the pores filled with an active mass. Alternatively, the construction is of pockets or tubes containing an active mass made conductive by the incorporation of a conductive alkali-resistant substance, such as nickel or cobalt. The nickel electrodes are sintered at about 900°C. for ½ hour. The proportion of powders of specified sizes to be used in making the plates is laid down.

SOCIÉTÉ DES ACCUMULATEURS FIXES ET DE TRACTION (Inventor: P. JACQUIER). *French Pat.* 1,011,115.

Electrodes for Gaseous Electric Discharge Devices

The electrodes consist of a relatively large prong or post of cold-rolled steel and a tip of an alloy of a refractory metal and an alkaline-earth metal: preferred composition:—tungsten 89, nickel 10, barium 1 per cent. The tip may be brazed to the body by a nickel-manganese brazing alloy.

BRITISH THOMSON-HOUSTON CO., LTD. *Brit. Pat.* 686,824.

Production of Electrolytic Nickel Anodes

Pure nickel anodes (containing copper 0·05-0·15, carbon 0·01-0·04, sulphur 0·1-0·15 per cent.) suitable for use in electrodeposition, are prepared from impure anodes by the following process:—

The impure anodes are electrolytically dissolved, and the electrolyte is purified from copper, arsenic, iron and lead. After such purification there is added to the electrolyte 0·1-0·15 g./L. of sulphur dioxide, 0·01-0·03 g./L. of copper (as a soluble copper salt), and 0·001-0·005 g./L. of acetylene. The electrodes are

then formed by cathodic deposition, without neutralizing the effects of the sulphur dioxide on the pH of the electrolyte.

L. S. RENZONI, assignor to INTERNATIONAL NICKEL CO., INC. *U.S. Pat.* 2,623,848 (continuation in part of 2,453,575).

Periodic-Reverse Plating in Electrodeposition of Nickel and Nickel-Cobalt Alloys

Bright electrodeposits of nickel, cobalt and nickel-cobalt alloys are produced by a periodic-reversed-current method. The electrolyte contains 0·1-0·005 oz./U.S. gallon of cadmium. The plating current flows for periods of not more than 2 seconds, and each reverse current pulse delivers at least 4 per cent. of the coulombs which passed in the preceding plating period: it preferably flows for ½ to 1/25th of the length of the plating period. A sulphate-chloride electrolyte and cadmium-containing anodes are preferred.

WESTINGHOUSE ELECTRIC INTERNATIONAL CO. *Brit. Pat.* 687,275.

Bright Nickel-Plating Baths

Sodium carboxymethylcellulose ('sodium cellulose glycollate') is used as a brightening agent in metal plating baths, e.g., in nickel and cadmium plating. Electrolytes claimed contain sodium cyanide, cadmium oxide and 0·1-2·5 per cent. of the brightening agent, or nickel sulphate, nickel chloride and boric acid, plus 0·05-0·10 per cent. of the brightener.

A. W. LIGER, assignor to WYANDOTTE CHEMICALS CORPN.

Plating of Sintered Materials

The electroplating of sintered materials, particularly sintered carbides, is facilitated, and adhesion of the coating is improved, by first blasting the sintered surface with fine abrasive grit, to remove carbon derived from the moulds. Typical examples relate to conditions for plating with nickel or cobalt.

D. NAPIER AND SON, LTD. (inventors: J. K. WILSON and O. WRIGHT). *Brit. Pat.* 685,734.

Nickel Coating of Propeller Blades

A composite aerofoil, e.g., a propeller blade, is formed of a light-metal base (aluminium), coated first with a tough adherent film of organic polymer and afterwards with a thin film of a corrosion- and erosion-resistant metal, preferably nickel, electrodeposited in a condition of expansive stress.

UNITED STATES RUBBER CO. *Brit. Pat* .684,012.

Electropolishing of Nickel-containing Materials

Stainless steel, other ferrous materials containing chromium and/or nickel, nickel and its alloys, and chromium and its alloys are electropolished in a bath based on a mixture of sulphuric and phosphoric acids, or in a sulphuric-acid solution, an addition being made, to both baths, of a relatively small amount of one or more aliphatic amino compounds. Preferred additives are benzylamine, triethanolamine, allylamine, or mixed isopropanolamines. The electrolyte may also contain a small amount of an aromatic amino compound.

P. A. CHARLESWORTH. *Brit. Pat.* 683,309.

See also—

Brit. Pat. 683,310 (also by P. A. CHARLESWORTH) covering the use of a heterocyclic compound or compounds having at least one heterocyclic ring containing nitrogen. Suitable compounds are indole, morpholine, and 8-hydroxyquinoline.

Nickel-containing Welding Rods for Welding Copper Alloys

Welding rods for joining copper and high-copper alloys consist of a core containing 50-100 per cent. nickel, balance copper, coated with (*a*) 20-60 per cent. by weight of a fluoride of barium, calcium or strontium, (*b*) 15-45 per cent. by weight of a carbonate of barium, calcium, or strontium, (*c*) 5-15 per cent. by weight of carbonaceous material, and 12·5-28 per cent. by weight of a solution of a silicate of sodium or potassium, serving as binder. The ratio of (*a*) : (*b*) : (*c*) is preferably 55:40:8.

EUTECTIC WELDING ALLOYS CO., LTD. *Brit. Pat.* 686,952.

Production of High-Permeability Nickel-Iron-base Alloys

In production of magnetic materials of the high-permeability type, the whole melting operation is conducted under a carbon-containing reducing slag, and the melt, after solidification, is decarburized by heat-treatment in hydrogen. Deoxidation is preferably by manganese only, the use of strong deoxidizers such as calcium, silicon, magnesium and aluminium being avoided. The procedure is specifically related to the production of alloys containing nickel 75-85, molybdenum 2-7, manganese up to 2 per cent., iron balance.

WESTERN ELECTRIC CO., INC. *Brit. Pat.* 681,466.

Production of Permanent Magnets having Columnar Structure

Permanent magnets characterized by unidirectional columnar crystals are cast by pouring the molten alloy into a mould cavity which has a transverse fin approximately at the mid-length of the cavity, and chilling the cavity at both ends. Sharp reduction in section in the middle of the casting prevents difficulties arising at the points where columnar crystals grown from the two opposite ends meet. The process is applicable to iron-base alloys containing aluminium +nickel, with or without cobalt and/or copper, and with or without small additions of numerous other elements.

DARWINS, LTD. *Brit. Pat.* 684,522.

Steel containing Spheroidal Graphite

The composition range of a steel containing spheroidal graphite is given as carbon 1·2-1·65, silicon 0·5-6, magnesium 0·03-0·5, sulphur less than 0·02, nickel 0-36, copper 0-3, manganese 0-2·5, chromium 0-2, molybdenum 0-2, per cent., balance iron. At least 0·35 per cent. carbon is preferably present as graphite, and the graphite may be wholly or predominantly

spheroidal as cast, or the steel may be cast with all or substantially all the carbon combined, and be subsequently heated above the critical temperature, to decompose the free massive carbides and to produce spheroidal graphite.

MOND NICKEL CO., LTD. *Brit. Pat.* 685,730.

Alloy Steel Shot

Steel pellets (e.g., for blast cleaning or peening) having good roundness and solidity, and free from dimples, cracks and strains, are made from steel containing carbon 0·10-1·7, silicon 0·30-1·0, manganese 0·30-2·0, chromium 0-5·0, molybdenum 0-5·0, vanadium 0-0·40, nickel 0-2·50, copper 0-1·0 per cent., balance iron.

AMERICAN WHEELABRATOR AND EQUIPMENT CORPN., assignee of T. J. HUTCHINSON.
Brit. Pat. 687,969.

Complex Nickel-Chromium-base Alloys for High-Temperature Service

Wrought austenitic precipitation-hardened alloys, particularly suitable for gas-turbine rotors, contain nickel 15-35, chromium 7-20, molybdenum and/or tungsten 2-5 total, titanium 1·3-1·9, aluminium not more than 0·4, carbon less than 0·1, manganese and/or silicon 0·3-3 per cent., iron balance. A suitable heat-treatment for forged parts of such alloys is solution heating at 1800°-2000°F. (980°-1095°C.), followed by ageing at 1300°-1450°F. (705°-785°C.).

G. R. SHEPHERD, communication from WESTINGHOUSE ELECTRIC INTERNATIONAL CO. *Brit. Pat.* 686,766.

Alloys suitable for gas-turbine parts, i.e., engine valves and other high-temperature components, contain carbon up to 0·15, silicon up to 2, manganese up to 2, nickel 28-32, chromium 21-24, cobalt 20-23, titanium 1·5-3 per cent., balance iron. Tungsten 2-6, molybdenum 2-6, boron up to 0·2, and aluminium 0·5-2·0 per cent. may also be present, provided that the combined tungsten and molybdenum content is between 2 and 10 per cent. The alloys are heat-treated as described in French Patent 1,004,175.

SOCIÉTÉ ANONYME DE COMMENTRY, FOURCHAMBAULT ET DECAZEVILLE.
French Patent of Addition 57,294 (addition to 1,004,175).

Mechanical Treatment of Heat-Resisting Austenitic Steel

In order to obtain good resistance to heat and creep, austenitic steels are cold worked until the elastic limit at the stress corresponding to a permanent elongation of 0·2 per cent. becomes equal to, or greater than, 45 kg./mm² (28·5 tons per sq. in.). The composition range for which such treatment is claimed as suitable is as follows:—carbon 0·05-0·15, chromium 16-18, nickel 11-14, silicon 0·1-1·0, manganese 0·2-1, tungsten 2·5-4·5, titanium 0·4-0·8, per cent.

SOCIÉTÉ D'ELECTRO-CHIMIE, D'ELECTRO-MÉTALLURGIE ET DES ACIÉRIES ELECTRIQUES D'UGINE.
French Pat. 1,008,515.

Mechanical and Thermal Treatment of Precipitation-Hardening Materials

Austenitic alloys of the precipitation-hardening type are heated to 2050°-2300°F. (1120°-1260°C.), subjected to a succession of plastic deformations and reheatings to the same temperature, and finally formed to shape at a temperature between room temperature and that of recrystallization. The product is then solution treated, preferably at 1700°-2300°F. (925°-1260°C.) for 5 to 50 hours. Such treatment is claimed to give enhanced mechanical properties at high temperatures and grain size controllable according to service requirements.

G. R. SHEPHERD, communication from WESTINGHOUSE ELECTRIC INTERNATIONAL CO.
Brit. Pat. 686,764.

Boron Additions to High-Temperature Alloys

Creep strength of alloys containing a large proportion of cobalt is increased by addition of small amounts of boron. The range claimed is carbon 0·05-1, silicon 0-2, manganese 0-2, nickel 10-40, cobalt 40-70, chromium 10-30, boron 0·001-0·5, vanadium 0-10, tungsten 0-3·5, molybdenum 0-3·5, niobium 0-5, tantalum 0-5, titanium 0-2, iron 0·5- 25 per cent. The following limitation is claimed (V+W+Mo±Nb+Ta+Ti)=3-15 per cent. Creep characteristics of a typical alloy at 800°C. are described.

WM. JESSOP AND SONS, LTD. (inventors: G. T. HARRIS and H. C. CHILD). *Brit. Pat.* 686,180.

Precipitation-Hardening Nickel-Chromium Steels containing Aluminium and Copper

Stainless steels capable of being precipitation-hardened after annealing and quenching contain chromium and nickel in balanced amounts (with aluminium, copper and silicon). Composition: nickel 4-6 per cent. at chromium 19 per cent., varying up to nickel 7·5-8 per cent. at chromium 14 per cent., together with carbon up to 0·15, aluminium 0·80-1·75, copper 2-5, manganese up to 2·0, silicon up to 2·0 per cent., iron balance. A preferred range of composition is cited.

H. TANCZYN, assignor to ARMCO STEEL CORPN.
U.S. Pat. 2,614,921.

Production of Stainless-Clad Steel

Stainless-clad ingots are produced by pouring mild steel containing at least 0·1 per cent. silicon into an ingot mould having one or more faces lined with stainless-steel plates, the inner surfaces of which have been cleaned from oxide.

RICHARD THOMAS AND BALDWINS, LTD., A. AGNEW and D. A. BISHOP. *Brit. Pat.* 683,330.

Corrigendum

Nickel Bulletin, Jan., 1953, p. 21, right-hand column, lines 2-4. For '5 per cent. of aluminium appears to be *sufficient* to prevent oxidation' please read '5 per cent. of aluminium appears to be *insufficient* to prevent oxidation'.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

THE MOND NICKEL COMPANY LIMITED
SUNDERLAND HOUSE · CURZON STREET · LONDON, W.1

PRINTED IN ENGLAND *by* WIGHTMAN MOUNTAIN LTD.
WESTMINSTER, LONDON, S.W.I